4044

CLINTON LABORATORIES
CENTRAL FILES NUMBER
47-3-439

RUN 15
(not very useful)

Ba 140 file

INV. 64

B-137

Date March 31, 1947

Subject Analytical Data Run # 15

Corrected to 2400, 1-27-47

By S. A. Reynolds

To L. B. Emlet

File C.7.

Those Eligible To Read The Attached

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L. B. Emlet

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1. L. B. Emlet
2. E. J. Witkowski
3. S. A. Reynolds
4. Central File

TO: L. B. Emlet
FROM: S. A. Reynolds

3-31-47

Analytical Data
Run #15
Corrected to 2400, 1-27-47

Step	Curies	Percent
Dissolving	2953	100.0
Extraction	520	17.6
Extraction cake washes	9	0.3
Metathesis	371	12.6
Metathesis cake washes	188	6.4
Electrolysis	203	6.9
Fuming nitric	13	0.4
HCl-ether	152	5.1
Miscellaneous tank rinses	282	9.5
TOTAL LOSS (KNOWN)	1738	58.8
FINAL PRODUCT IN B-6	764	25.9

Material Balance 84.7%

3-3

Ba^{140} file

CLINTON LABORATORIES
~~CENTRAL FILES NUMBER~~
47-3-80

R. I.

B-137

Date March 4, 1947

Subject Ra-La Analysis

By L. B. Emlet

To R. W. Spence

File c.7.

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Date AUG 24 1971

RUN 15A

140 Br file

CLINTON LABORATORIES
CENTRAL FILES NUMBER
47-3-89

R.L.

B-137

File C.7.

Date March 4, 1947

Subject Analytical Data Run #15A (Shipment # 23)

By S. A. Reynolds

To L. B. Emlet

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L. B. Emlet

INV. 64

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TO: L.B. Emlet
FROM: S.A. Reynolds

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By Authority Of
By [signature] Date AUG 24 197

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Single rereview of CCRP-declassified documents was authorized by DOE Office of Declassification memo of August 22, 1994.

Analytical Data
Run #15A
(Shipment #23)
L.S.T. 0537, 3-4-47

Dissolvings / **Extraction Wastes**

Batch	Curies	Slugs		A8	A6	Act. Loss
A	301	65	294 loaded?	9	—	0.3
B	309	72		371	10	0.4
C	267	60		51	13	0.5
D	385	75	300 loaded	97	11	0.5
E	379	69		565	17	0.6
F	354	67		94	18	0.6
G	309	62		164	19	0.7
H	325	67		68	—	2.4
I	154	32		36	—	1.3
Totals	2783	569	594	Act. Loss	204	7.3

	Curies	Per Cent
Other Cell A Loss		
Washes of Ext. Cake	26	0.9
Metathesis	209	7.5
Metathesis Cake Wash	32	1.2
A9 Rinse	31	1.1
Total Cell A Loss	502	10.0
Material Balance Through Electrolysis		
Electrolysis loss	15	0.5
Product in B6	2081	74.8
Material Balance 93.3%		
Cell B Losses		
Electrolysis	15	0.5
B6 Rinse	8	0.3
Fuming Nitric Waste	33	1.2
HCl-Ether Waste	130	4.7
Total Cell B Loss	186	6.7
Total loss (Known)	688	24.7
Product in B17	1925	69.2
Material Balance 93.9%		
B17 Rinse	13	

Product Activity Shipped 1900 (Approx)

Spectrographic Analysis of Product
(1) Pb, less than 4mg; (2) Fe, less than 0.8 mg; (3) Cr, 2mg; (4) Ni, 4mg, (5) Sr, 1.6 mg.; (6) Ba, 290 mg.

Radiation Reading ("Skyshine")
22r/hr at 20 hr. after L.S.T. indicating about 1850 c.

[redacted]

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M-208

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[redacted]

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2. E. J. Witkowski
3. L. B. Emlet
4. Reading File
5. Central File

CLINTON LABORATORIES
CENTRAL FILES NUMBER
47-4-96
c. 7.

MONSANTO CHEMICAL COMPANY

CLINTON LABORATORIES
P. O. BOX 1991
KNOXVILLE 11, TENNESSEE

April 4, 1947

Classification Cancelled
[redacted]
By Authority Of
By C S B Date AUG 25 1971

INV. 64

Dr. R. W. Spence
P. O. Box 1663
Santa Fe, New Mexico

Dear Dr. Spence:

The following is the analysis of Batch 16 17 of Ra-La shipped from Clinton Laboratories on April 4, 1947:

1020 C - LST - 7-30 ~~2456~~ 2478 C - LST

Pb	-	3.9 mg -	3.0 mg
Ni	-	7.8 mg	7.0
Cr	-	5.8 mg	.7
Ba	-	223 mg	350
Fe	-	5.8 mg	7.0
Sr	-	0.01 mg.	2.0

LST = 2045 - April 2, 1947. - ~~0500 - 4/29/47~~
LST = 0500 - EST - 4/29/47

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L B Emlet
L. B. Emlet

LBE:wq

#1326

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CLINTON LABORATORIES
CENTRAL FILES NUMBER
47-4-204

Ba 140 file

RUN 16

R.I.

B-137

Date April 11, 1947

File C.7.

Subject Analytical Data, Run #16(Shipment #24)

L.S.T. 2045, 4-2-47

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By S. A. Reynolds

Copy # 2A

L. B. Emlet

To L. B. Emlet

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April 11, 1947

TO: L. B. Emlet
FROM: S. A. Reynolds

[Classifi]cation Cancelled or Changed
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By Authority of
AUG 31 1971
Name Title Date

Analytical Data
Run #16
(Shipment #24)
L.S.T. 2045, 4-2-47

DECLASSIFIED

Dissolvings			Extraction Wastes		
Batch	Curies	Slugs	A5	A6	% Act. Loss
A	[illegible]	62	11	—	0.4
B	223	61	157	7	0.3
C	218	58	235	9	0.3
D	332	70	136	13	0.5
E	339	69	120	17	0.6
F	3[illegible]	73	40	—	1.[illegible]
G	230	59	151	18	0.[illegible]
H	281	63	41	—	[illegible]
I	258	60	247	5	[illegible]
J	180	45	Act. Loss	119	4.[illegible]
Total	2667	620	Act. Loss	280	10.5

Cell A Loss	Curies	Per Cent Act. Loss
[illegible] Cake (1)	719	
[illegible]	6	0.2
[illegible]	141	5.4
[illegible] Cake Wash	131	4.9
[illegible] 19	3	0.1
[illegible] in Cell A (Calc. from an A9 sample)	565	21.2
Total Cell A Loss	1128	42.3
Material Balance Through Electrolysis		
Electrolysis Loss	6	0.2
Product in [illegible]	1486	55.7
Material Balance 98.2 %		
Cell B Losses		
Electrolysis	6	0.2
[illegible]	4	0.1
[illegible] Waste	37	1.4
[illegible] Waste	121	4.5
Total Cell B Loss	168	6.3
[illegible]	1296	48.6
[illegible]	1020	38.2
[illegible] 86.8%		
[illegible]	32	
Product Shipped	990 Approx.	

Spectrographic Analysis of Product
(1) [illegible], 4mg.; (2) Fe, 6mg., (3) Cr, 6mg. (4) Ni, 8mg.; (5) Sr, 0.1 mg.;
(6) Ba, 223 mg.

Radiation Reading ("Skyshine")
About 10 r/hr at 20 hr after L.S.T., indicating about 840 c.

CLASSIFICATION [illegible]
ADD signature
Single review of [illegible]
documents was authorized [illegible]
Declassification [illegible]

[redacted]

[redacted]

RUN 17

CLINTON LABORATORIES
CENTRAL FILES NUMBER
47-5-73
c.7

B-137

R. I.

Date May 1, 1947

Subject Analytical Data

File

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By S. A. Reynolds

INV. 64

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L. B. Emlet

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~~May~~ 1, 1947

TO: L.B. Emlet
FROM S.A. Reynolds

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Analytical Data
Run # 17
(Shipment #25)
L.S.T. 0500, 4-29-47

Dissolvings			Extraction Wastes		
Batch	Curies	Slugs	A8	A6	% Act. Loss
A	304	74	48	-	1.3
B	273	67	108	17	0.5
C	223	60	75	6	0.2
D	295	66	114	6	0.2
E	290	64	338	12	0.3
F	301	66	804	6	0.2
G	268	62	957	10	0.3
H	231	55	198	6	0.2
I	358	61	998	8	0.2
J	340	64	1659	6	0.2
K	324	65	287	7	0.2
L	328	64	174	8	4.9
Totals	3535	768	Act. Loss	306	8.7

	Curies	Per cent
Other Cell A Loss		
Wash of Ext. Cake	10	0.3
Metathesis	177	5.0
Metathesis Cake Wash	49	1.4
Left in cell A Loss	2	—
Total Cell A Losses	544	15.4
Cell B Losses		
Electrolysis	15	0.4
B6 Rinse	27	0.8
Fuming Nitric Waste	122	3.5
HCl-Ether Waste	137*	3.9
Total Cell B Loss	301*	8.5
Total Loss	845	23.9
Product in B17	2478	70.1

Material Balance 94.0%
* Assumed. HCl-Ether sample was bad.

CLASSIFICATION CANCELLED
Giles Morgan 12-29-94
ADD signature Date
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[redacted]

Spectrographic Analysis of Product
(1) Pb, less than 3 mg.; (2) Fe, 7 mg.; (3) Cr, 0.7 mg.; (4) Ni, 7 mg.; (5) Sr, 2mg.; (6) Ba, 350 mg.

Radiation Reading ("Skyshine")
About 29 r/hr. at 20 hr. after L.S. T., indicating about 2440 curies.

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CLINTON LABORATORIES
CENTRAL FILES NUMBER
47-6-304

Run 18

B-137

R.I.

File C.7.

Date June 19, 1947

Subject Analytical Data Run #18 (Shipment #~~24~~ 26.)

INV. 64

By S. A. Reynolds

To L. B. Emlet

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TO: L.B. Emlet
FROM: S.A. Reynolds

June 19, 1947

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Analytical Data
Run #18 W
(Shipment #24)
L.S.T. 0545, 6-18-47

Dissolvings

Batch	Curies	Slugs	A8	A6	% Act. Loss
A	290	66	56	1	0.03
B	255	63	66	7	0.20
C	242	62	216	4	0.11
D	305	69	145	1	0.03
E	237	59	273	64	1.85
F	268	69	44	8	0.23
G	259	68	71	5	0.14
H	308	70	195	10	0.30
I	276	68	476	6	0.17
J	258	62	83	7	0.20
K	250	67	107	2	0.06
L	255	65	476	19	0.55
M	249	61	130	—	3.76
	3452	849	Act. Loss	264	7.64%

Other Cell A Losses

	Curies	Per Cent
Washes of Ext. Cake	284	8.2
Metathesis	97	2.8
Metathesis Cake Wash	329	9.5
Total Cell A Loss	982	28.4

Material Balance Through Electrolysis

Electrolysis Loss		14	0.4
Total Loss		996	28.8
Product in B-6		2393	69.3
Material Balance	98.1%		

Radiation Reading ("Skyshine")
About 20r/hr at 9 hours after L.S.T. indicating approximately 2330 curies.

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Date AUG 25 1971

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CLINTON LABORATORIES
CENTRAL FILES NUMBER
47-7-380

RUN 19

B-13

B-137

Completed

R.L.

INV. 64

File C.7.

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Copy # 1
L. B. Emlet

Date July 25, 1947

Subject Analytical Data Run # 19, Shipment # 27, L.S.T. 2140, 7-21-47

By S. A. Reynolds

To L. B. Emlet

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July 25, 1947

To: L.B. Emlet
From: S.A. Reynolds

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Date AUG 25 197

Analytical Data
Run # 19
(Shipment # 27)
L.S.T. 2140, 7-21-47

Dissolvings

Batch	Curies	Slugs	A-8	A-6	Per cent Act. Loss
A	256	65	75	2	0.06
B	237	63	170	2	0.06
C	253	65	48	5	0.16
D	257	68	415	7	0.23
E	229	64	242	8	0.25
F	254	64	143	22	0.71
G	214	59	421	29	0.94
H	259	70	1054	13	0.42
I	221	65	957	18	0.58
J	260	70	1541	13	0.42
K	234	65	202	15	0.49
L	214	65	170	7	0.23
M	199	59	209	—	6.77
	3087	842	Act. Loss	350	11.33

Other Cell A Losses	Curies	Per cent loss
Washes of Ext. Cake	3	0.10
Metathesis Cake and wash	321	10.40
B-1 Rinse	3	0.10
A-9 Rinse	32	1.04
Total Cell A. Loss	709	22.97
Cell B Losses		
Electrolysis	25	0.81
B-6 Rinse	9	0.29
Fuming Nitric Waste	6	0.19
HCl-Ether Waste	132	4.28
Total Cell B Loss	172	5.57
Total Loss	881	28.54
Product in B-6	1785	57.82
Material Balance	86.36%	

Radiation Reading ("Skyshine")
21.4 r/hr at 20 hours after L.S.T. indicating Approximately 1800 Curies.

This [redacted]

RUN 20 B-13

B-137

47-8 329

INV 77

Completed

File Anal.

Date August 19, 1947

Subject Analytical Data Run #20, (Shipment #28)

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By J. Davidson

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To

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INV 59

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47-8 329

Analytical Data
Run #20
(Shipment#28)
L.S.T. 0530 8-19-47

Batch	Curies	Slugs	A-8	A-6	% Act. Loss
A	233	63	44	—	1.30
B	218	61	25	4	0.12
C	212	58	158	6	0.18
D	238	68	80	11	0.32
E	218	63	111	7	0.21
F	223	63	92	8	0.24
G	199	60	305	12	0.35
H	280	67	193	7	0.21
I	283	67	47	—	1.39
J	273	69	62	7	0.21
K	240	62	98	9	0.26
L	269	66	258	17	0.50
M	265	67	191	14	0.41
N	239	64	304	—	8.97
	3390	898	Act Loss	497	14.67

Other Cell A Losses	Curies	Per cent Loss
Washes of Ext. Cake	17	0.50
Methathesis	151	4.45
Methathesis Cake Wash	48	1.42
A-9 Rinse	47	1.39
Total Cell A Loss	760	22.43

Cell B Losses	Curies	Per cent Loss
Electrolysis	10	0.29
B-6 Rinse	20	0.59
Fuming Nitric Washe	14	0.41
HCl-Ether Waste	94	2.77
Total Cell B Loss	138	4.07
Total Loss	898	26.50

Product

Based on Radiation Reading ("Skyshing")	2700	79.64
Material Balande	106.14%	

Classification Cancelled
Or ~~Changed To~~ EKAa
By Authority Of NR
By [redacted] Date SEP 12 1977
M. Day

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T. Central file

RUN 21

CLINTON LABORATORIES
CENTRAL FILES NUMBER
47-10 630

B-137

Date October 1947, 19

File XT

Subject Analytical Data Run #21 (Shipment #29)

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By James Davidson

To

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Central Files

CLINTON LABORATORIES
CENTRAL FILES NUMBER
47-10 630

Analytical Data
Run #21
(Shipment #29)
L.S.T. 1410 10-19-47

Batch	Curies	Slugs	A-8	A-6	% Actual Loss
A	200	66.0	—	4	0.09
B	238	78.0	—	4	0.09
C	184	58.0	—	7	0.16
D	253	71.0	—	9	0.20
E	207	68.0	18	5	0.11
F	171	56.0	42	12	0.27
G	205	70.0	—	13	0.29
H	222	70.0	8	—	0.18
I	211	70.0	8	—	0.18
J	205	65.0	37	—	0.84
K	184	65.0	26	—	0.59
L	183	64.0	43	—	0.97
M	194	60.0	574	6	0.14
N	328	71.0	33	18	0.41
O	332	66.0	5	—	0.11
P	279	59.0	60	52	1.18
Q	285	61.0	703	13	0.29
R	296	66.0	42	—	0.95
S	221	57.0	—	—	—
T	24	7.0	—	—	—
	4422	1248.0	Actual Loss 312		7.05

Other Cell A Losses	Curies	% Loss
Washes of Ext. Cake	169	3.82
Metathesis Cake Wash	121	2.74
A-9 Rinse	39	0.88
1MS & 1MT Heels	247	5.59
Rinse of Cell A Tanks (A-6 Tank Sp.)	408	9.23
Total Cell A Loss	1296	29.31
Cell B Losses		
Electrolysis	64	1.45
B-6 Rinse	12	0.27
Fuming Nitric Waste	18	0.41
HCl-Ether Waste	232	5.25
Rinse of B26 & B27	54	1.22
Total Cell B Loss	380	8.59
Total Loss	1676	37.90
Product		
Product in B-6	2612	59.06
Material Balance	96.96%	

Radiation Reading

From "Y": Radiation reading indicates approximately 2400 curies.

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By Authority Of
By
Date
AUG 30 1971

[redacted]

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ADD signature
Date 1/9/95

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[redacted]

RUNS 22 & 22A

B-13

CLINTON LABORATORIES
CENTRAL FILES NUMBER
47-12-233

Classification Cancelled
B-137
By Authority of DOC
By [signature] Date AUG 9 1973

Completed

Date December 9, 1947

File Anal.

Subject Analytical Data Ba Runs #22 and 22A (Shipments #30 and #31)

Those Eligible To Read The Attached

By S. A. Reynolds

Copy # 1 A

To

L. B. Emlet

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Analytical Data
Ba Runs #22 and #22A.
(Shipments #30 and #31)
L.S.T. 1200, 11-26-47

CLINTON LABORATORIES
CENTRAL FILES NUMBER
47-12- 232
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Batch	Curies	Slugs	A-8	A-6	% Actual Loss
A	185	64.8	5	—	0.12
B	184	67.9	—	3	0.08
C	175	66.5	10	—	0.25
D	188	70.2	5	—	0.12
E	179	66.0	11	—	0.28
F	174	68.7	4	—	0.10
G	153	60.7	9	—	0.23
H	192	67.4	39	8	0.20
I	172	78.0	2	—	0.05
J	197	69.8	24	2	0.05
K	167	65.4	57	5	0.12
L	244	78.0	108	6	0.15
M	197	65.6	87	14	0.35
N	195	67.0	13	—	0.33
O	156	57.2	17	—	0.42
P	245	69.0	31	—	0.78
Q	253	67.7	65	15	0.37
R	197	60.0	15	—	0.37
S	204	68.2	27	—	0.68
T	166	53.4	82	—	2.05
U	172	57.7	19	—	0.48
	3995	1389.0	Actual Loss	303	7.58%

Other Cell A Losses	Curies	% Loss
Washes of Ext. Cake	32	0.80
Metathesis	63	1.58
Metathesis Cake Wash	25	0.63
A-5 Sump	17	0.43
A-9 Rinse	49	1.23
Total Cell A Loss	489	12.25
Cell B Losses		
Electrolysis	45	1.13
B-6 Rinse	16	0.41
Fuming Nittic Waste	55	1.38
HCl-Ether Waste	147	3.68
Total Loss (Through B-6)	534	13.37
Product		
Product in B-6		
* Shipment #30	1495	37.4
" #31	1321	33.1

Material Balance (thru B-6) 83.87%

* This shipment made 11-21-47

[redacted]

Please ... C.F. -

RUN 23

B-137

Anal. Data on Runs

Date January 28, 1948

File ________

Subject Analytical Data Ba Run 23, Shipment #32

Those Eligible To Read The Attached

By S. A. Reynolds

Copy # 1A

To L. B. Emlet

~~L. B. Emlet~~

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1-28-48

Analytical Data
Ba Run 23
Shipment #32
L.S.T. 1500, 1-22-48

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No. 1 of 4 copies, Series A.

CLINTON LABORATORIES
CENTRAL FILES NUMBER
48-1-405

To: L.B. Emlet
From: S.A. Reynolds

Batch	Curies	Slugs	A-8	A-6	% Actual Loss
A	282	68.8	58	2	0.06
B	313	84.1	25	----	0.73
C	270	76.3	55	7	0.20
D	261	69.8	43	----	1.26
E	301	72.0	20	----	0.59
F	264	64.7	242	11	0.32
G	248	75.0	207	12	0.35
H	218	69.1	450	10	0.34
I	274	77.1	15	----	0.44
J	277	76.9	218	12	0.35
K	307	75.4	79	10	0.34
L	237	68.0	7	5	0.15
M	155	43.1	411	13	0.38
N-Heels	4	---	---	----	----
	3411 ✓	920.3	Actual Loss	185	5.42%

Other Cell A Losses	Curies	% Loss
Washes of Ext. Cake(8.W-3 & 6W-1)	91	2.67
Metathesis	69	2.02
Metathesis Cake Wash	33	0.97
A-9 Rinse	6	0.17
LMN Heels	4	0.12
Total Cell A Loss	388	11.37% ✓

Cell B Losses		
Electrolysis	16	0.47
B-6 Rinse	16	0.47
Fuming Nitric Waste	29	0.85
HCl-Ether Waste	91	2.67
Total Loss (Cell B)	152	4.46% ✓

Product		
Product in B-6	3016	88.4 % Product
Shipment #32		
Material Balance (through B-6) 99.8%		

Radiation Reading ("Skyshine")

Indicates approximately 2750 curies

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CENTRAL FILES NUMBER

48-3-307

RUN 24

Mat. Gen. B.

B-137

File

Date March 12, 1948

Subject Analytical Data Re Run #24

Shipment #33

By E. J. Witkowski

To

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CENTRAL FILES NUMBER
48-3-307

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Analytical Data
Ba Run #24
(Shipment #33)
L.S.T. 1030, 3-12-48

L.B. Emlet
E.J. Witkowski
S.A. Reynolds
Central Files

Batch	C's	Slugs	A-8	A-6	% Loss	Material Balance
A	199	65.5	2	---	0.05	197
B	180	68.0	2	---	0.05	375
C	167	60.7	20	---	0.50	522
D	210	72.3	28	---	0.65	704
E	192	68.8	51	5	0.12	891
F	183	67.0	50	9	0.21	1065
G	180	72.0	79	6	0.14	1239
H	179	66.8	61	6	0.14	1412
I	203	70.5	37	12	0.28	1603
J	178	66.3	219	11	0.26	1770
K	195	70.4	33	11	0.26	1954
L	174	60.7	60	8	0.19	2120
M	124	50.2	24	5	0.12	2239
N	92	27.3	6	---	0.19	2323
O	324	66.1	122	7	0.16	2640
P	290	60.4	90	28	0.65	2902
Q	262	64.3	71	18	0.42	3146
R	304	60.0	71	11	0.26	3439
S	314	63.6	560	4	0.09	3749
T	269	58.6	683	15	0.35	4003
A-1					0.---	
Heel	57	13.3				4060
	4276	1272.8	Actual Loss	216	5.05% Loss	

Other Cell A Losses	Curies	% Loss	Material Balance
Washes of Ext. Cake			
SWW-B	108	2.52	3952
Metathesis SWC-B	36	0.84	3916
Metathesis Cake Wash			
SWCW-A	61	1.43	3855
A 9 Rinse	63	1.47	3792
A 1 Heels	57	1.33	3735
Total Cell A Loss	541	12.65%	
Cell B Losses			
B6 (Lead removal)	48	1.12	3687
B12 (Lead Removal)	2	0.05	3685
Electrolysis			
SWPb A	4	0.09	3681
SWPb B	20	0.50	3661
B6 Rinse	11	0.26	----
Fuming Nitric Waste	36	0.84	----
HCl-Ether Waste	127	2.97	
Total Loss	248	5.80%	

Product
Product in B6 2900 c's 67.8% Product
Material Balance (Thru B6) 79.2% Material Balance
Radiation Reading (Skyshine) indicates 3112 C's (or 4170 L.A. Units)

[redacted]

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By [illegible]
Date AUG 6 1971

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B-137

RUN 26

Date July 26, 1948

Subject Analytical Data Be Run #26

Shipment #34

By E. L. Wyatt

To L. B. Emlet

File C-7.

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L. B. Emlet

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To Tom Widner	From Bob Burns
Co./Dept.	Co.
Phone #	Phone #
Fax #	Fax #

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No. 1 of 4 copies, Series A
7-26-48

To: L.B. Emlet
From: E.I. Wyatt

Analytical Data
Ba Run #26
(EST 1520 - 7-21-48)
Shipment #34

OAK RIDGE NATIONAL LABORATORY
CENTRAL FILES NUMBER
48-7-326

Batch	Curies	Slugs	A-8	A-6	% Loss	Material Balance
Residuals from Run 25	345	58.5	---	---	---	345
A	239	70.7	35	---	0.78	549
B	235	72.7	121	7	0.16	777
C	220	57.0	49	---	1.09	948
D	276	77.6	73	15	0.33	1209
E	275	72.7	546	9	0.20	1475
F	277	78.9	75	15	0.23	1737
G	259	72.2	244	10	0.22	1986
H	280	76.2	59	11	0.24	2255
I	289	77.5	81	12	0.27	2532
J	324	75.1	427	8	0.18	2848
K	275	73.7	430	5	0.11	3118
L	262	78.4	176	---	3.91	3204
M	245	70.6	---			3449
Heel Diss. #2	269	71.1	---			3718
Heel Diss. #3	223	66.5	---			3941
Heel Diss. #4	174	57.0	---			4115
Heel Diss. #5	34	8.6	---			4149
Total	4501	1215	Actual Loss	352	7.82	4149

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By

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Other Cell A Losses	Curies	% Loss	Material Balance
Wash of Extract. Cake (SWW)	16	0.36	4133
Metathesis (SWC)	181	----	----
Metathesis Cake Wash (SWCW)	145	3.22	3988
A9-Rinse	6	0.13	3982
Al-Heels	700	15.55	3282
Batch M	245	5.44	3037
Total Cell A Losses	1464	32.52	

Cell B Losses	Curies	% Loss	Material Balance
Electrolysis (SWPb)	25	0.56	3012
B6 Rinse	11	0.24	
Fuming Nitric Waste	17	0.38	
17 HCl-ether Waste	131	2.91	
Total Cell B Loss	184	4.09	

	Curies	% Loss	Material Balance
Total Losses	1648	36.61	

Product			
Product in B6 (Radiochemical)	2588	57.5%	
Material Balance (thru B-6)		90.58	
Total Product Shipped (Radiochemical)	2429	53.97	
Product shipped (Skyshine readings)	2419	53.74	3387 LA Units
Material Balance by skyshine		90.36	

cc:
E. Witkowski
Central Files

[redacted]

[redacted] B-03

OAK RIDGE NATIONAL LABORATORY
CENTRAL FILES NUMBER
48-9-40

RUN 27

B-137

Date September 6, 1948

File C.7.

Subject Analytical Data, Be Run #27.

Shipment #35

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By E. I. Wyatt

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To L. B. Emlet

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L. B. Emlet

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To: L.B. Emlet
From: E.I. Wyatt

Analytical Data
Ba Run #27
L.S.T. 0820-9-3-48
Shipment #35

Date 9-6-48

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OAK RIDGE NATIONAL LABORATORY
CENTRAL FILES NUMBER
48-9-40

Batch	Curies	Slugs	A-8	A-6	% Loss
A	245	57.3	8	---	00.3
B	228	62.5	15	---	00.5
C	206	54.2	16	---	00.5
D	255	62.5	26	---	00.8
E	225	60.3	23	---	00.7
F	215	57.6	146	21	00.7
G	195	60.2	201	34	01.1
H	248	63.4	49	---	01.6
I	209	69.8	95	17	00.6
J	220	58.8	69	18	00.6
K	201	58.1	64	32	01.0
L	186	55.6	110	37	01.2
M	246	61.7	216	11	00.4
N	196	52.6	91	---	02.9
O (Heels)	12	3.1	---	---	---
Total	3087	837.7	Actual Loss	398	12.9%

Other Cell A Losses	Curies	% Loss
Wash of Ext. Cake (8WW)	9	00.3
Metathesis (8WC)	63	----
Metathesis Cake wash (8WCW)	74	02.4
A-9 Rinse	81	02.6
Al-Heels (1MO)	12	00.4
Total Cell A Losses	574	18.6

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TO
By Authority of
ATG AUG 26 1971
Name Title Date

Cell B Losses	Curies	% Loss
Electrolysis (5WPb)	20	00.6
B6-Rinse	5	00.2
Fuming Nitric Waste	24	00.8
HCl-ether Waste	174	5.6
	223	7.2
Total Losses	797	25.8

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Product

Product in B6 Radiochemical	2275	------
Material Balance (through B6) %	----	92.9
Total Product shipped (Radiochemical)	2072	
Product shipped (Skyshine Readings)	2196	
Or LA Units	3075	
Material Balance through Skyshine		97.0

[redacted]

cc:
E. Witkowski

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B-13

B-137

RUN 28

OAK RIDGE NATIONAL LABORATORY
CENTRAL FILES NUMBER
48-11-230

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Date November 23, 1948

Subject Analytical Data Ba Run #28

Shipment #36

By E. I. Wyatt

To L. B. Emlet

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To: L. B. Emlet
From: E. I. Wyatt

Analytical Data
Ba Run #28
L.S.T. 1310-11-21-48
Shipment #36

Date 11-23-48

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Batch	Curies	Slugs	A-8	A-6	% Loss
A	2319	32.7	319	20	0.7
Al Heels	300	3.6			
Total	2619	36.3	---	20	0.7

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Other Cell A Losses

	Curies	% Loss
Wash of Ext. Cake (8WW)	139	5.3%
Metathesis (8WC)	169	---
Metathesis Cake Wash (8WCW)	214	8.2%
A-1 Heels	300	11.5%
A-9 Rinse	8	0.3%
Total Cell A Losses	681	26.0%

Cell B Losses

	Curies	% Loss
Electrolysis (3WPb)	19	0.7%
B-6 Rinse	7	0.3%
Fuming Nitric Waste	21	0.8%
HCl-Ether Waste	154	5.8%
Total Cell B Loss	201	7.6%

	Curies		% Loss
Total Losses	882		33.6%
			13.0
			46.6

Product

	Curies		%
Product in B-6 (Radiochemical)	1641		62.7%
Material Balance (through B-6)	----		89.4%
Total Product Shipped (Radio chemical)	1459		
Total Product Shipped (Skyshine)	1380	(LA Units-1930)	52.7%
Material Balance through Skyshine	----		86.4%

cc: E. Witkowski
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48-12-192

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DATE December 15, 1948

SUBJECT RaLa Production from Hanford Slugs and Product Contamination Studies

COPY NO. 6 A

TO C. N. Rucker

FROM E. J. Witkowski and L. B. Emlet

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RALA PRODUCTION FROM HANFORD SLUGS AND PRODUCT CONTAMINATION STUDIES

December 15, 1948

Rala Run #28, the first in which Hanford-irradiated slugs were used, was very successful from the standpoint of our processing. No unusual difficulties were encountered and only minor changes in the old process were required.

The quantity of product produced and shipped was only 1,380 curies while at least 2,000 curies were anticipated. The chemical analyses of the dissolver solution indicated that the thirty-eight, four-inch slugs used contained only 4,500 curies at the time of pile discharge while the original calculations indicated that over 10,000 curies should have been present providing that the pile operated normally and the slugs were exposed at maximum neutron flux. This lack of product was at first thought to be a result of the abnormal operation of the Hanford "B" pile. In checking with Hanford, however, we learned that their calculations, which take into consideration the abnormal operation of the pile, indicate that 8,100 ±5% curies were present at the time of discharge. The reason for this discrepancy in curie content is not known. We do feel certain, however, that the errors in our analyses could not contribute materially toward explaining the difference; all of the analyses made during processing checked well, the material balance was normal, and the several radiation readings on samples and at the entrance to the cell showed a low quantity of product. We are requesting Hanford to review their calculations in an attempt to explain this disagreement.

Under normal circumstances, material from our pile could have been used to increase the amount of product finally isolated. Unfortunately it was not practical at this time because of the excessive pile-down time due to the installation of the new exhaust air filter units.

Even though the quality of these slugs was much poorer than anticipated, their use was proven to be economical. The run was completed in five days as compared to the usual ten-day period, waste solutions were of low volume and more concentrated product solutions resulted. We intend to use them in the next run scheduled for January 10, 1949, when it is hoped that more information will be obtained which will offer an explanation of what happened to the slugs used in Run #28.

[redacted]

Witkowski and Emlet to Rucker
December 15, 1948

RaLa Production from Hanford Slugs and Product Contamination Studies

On November 8 and 9, 1948, Mr. Leary and Drs. Mueller and White, all of Los Alamos, visited us to discuss the possibility of eliminating some of the contaminants from our product. They felt that the impurities, suspected to be organic matter, were contributing a great deal to the product losses encountered during processing at Los Alamos. These losses also resulted in radiation exposure to their operating personnel.

During our conversations, various potential sources of contamination were discussed and it was decided that the following points would be investigated:

1. A dirty product evaporation head heater. This head heater is exposed to the cell atmosphere between runs and it appeared possible that some dirt was collected on this heater which was washed into the product during evaporation.

2. Refluxing of vapors in the product evaporation off-gas line. It was thought that such action would result in washing back of stainless steel corrosion products into the product.

3. Polymerization of the ether or alcohol, or the impurities present in these solvents which are used in the purification process.

4. The Tygon tubing used for solution addition lines and all transfer lines to and from the glass purification equipment.

Each of the above four sources of potential contamination were investigated as follows:

1 & 2. An attempt was made to correlate the length of time during which the product evaporation equipment was idle before each run and the quality of product as reported by Los Alamos. It was thought that the amount of dirt which might accumulate on the head heater would depend on the time it was idle. No results were obtained from this comparison.

[redacted]

[redacted]

1 & 2. Continued

In addition, two dummy evaporations were made using an inactive solution of barium chloride. Both runs turned out to be very clean, much cleaner than the usual product; no organic material was found in either of these runs.

These dummy runs made it evident that the product evaporation operation was not the main source of product contamination. The possibility of a dirty head heater causing any difficulty was also shown to be unlikely since the product of the first dummy run was clean in spite of the fact that the heater had been idle for several months before the run was made.

3. Because it is almost impossible to duplicate the extreme radiation to which the HCl-ether and HCl-alcohol solutions are subjected during a regular run, no conclusive results are available to indicate the possibility of product contamination by polymerization of these reagents or their impurities. From the data available, there appears to be no relationship between the length of time required for the filtration of the HCl-ether or HCl-alcohol wastes (time of contact between solution and product) and the quality of the product reported by Los Alamos. This problem is being investigated further.

4. The possibility of contamination by Tygon tubing was investigated by making two dummy runs through the entire fuming nitric and HCl-ether purification and the product evaporation processes. All conditions in the first run were identical to those of a regular run except for the absence of radiation. The product from this run had a tan color similar to that of regular runs. When $BaCl_2$ was dissolved, it was found to contain some insoluble foreign matter composed of approximately ten percent carbon.

The second run through the purification steps was made after the Tygon solution addition line was replaced with an MFP-10 plastic tube which is inert to practically all reagents. No other tubes could be replaced at that time because of the limited amount of time before the start of Run #28. This dummy run turned out much cleaner than the first. Upon dissolving of the product very little foreign material was found; not enough was available for a carbon analysis.

[redacted]

4. Continued

These two dummy runs plus some laboratory tests run on Tygon tubing itself indicate that Tygon contributed, at least a portion of the contaminants found in the product. The laboratory tests show that Tygon is not very resistant to the reagents used in the purification process. It is nitrated by the fuming nitric acid and the nitrated material is soluble in ether.

As previously mentioned, no attempt was made to replace more than just the solution addition line before the start of Run #28. The only other change made in the purification steps was that specially-purified ether and HCl were used instead of the C.P. grade normally used. The alcohol for product washing was also eliminated.

The report from Los Alamos on the product of Run #28 was disappointing. They report that the product was dirty and that they lost a large portion of it because of the difficulty encountered in dissolving it from the shipping cone. They think that this difficulty was caused by what appeared to be a drop of oil on the shoulder of the cone. This we cannot explain. They did not experience a high loss in the hydroxide precipitation as they normally do (probably because they filtered the initial solution in HCl), but they did get a "crud" formation which gave them trouble during the oxalate precipitation and filtration.

Since the only source of contamination evident at the present time is the Tygon tubing, we have decontaminated our small cells in preparation for the replacement of all Tygon tubing wherever possible. We believe that it will be possible to eliminate Tygon completely in one of the duplicate sets of glassware equipment. The other set of equipment will have at least all of the Tygon product transfer lines and solution addition lines replaced. As an added precaution, purified reagents will be used in the purification process.

In addition to these immediate corrective measures, plans are now being made to investigate the effects of radiation on ether and oxalates. Judging from our limited experience with oxalates we suspect that they break down under beta-gamma radiation. It is only a guess but this may account for at least a portion of the troubles encountered at Los Alamos with the last shipment.

We have suggested that Los Alamos continue the filtration of their initial solution of our product. Our dummy runs indicate that the removal of the "crud" at this stage is relatively simple and should prevent interference later in the process.

[redacted]

Witkowski and Emlet to Rucker
December 15, 1948

Rala Production from Hanford Slugs and Product Contamination Studies

At the same meeting with the Los Alamos representatives, the quantity of inert barium (from Hanford slugs) which could be expected in 10,000 and 20,000-curie shipments was also discussed. The results of our calculations, made on the assumption that the existing process would be used, is indicated in the table below. The slug-irradiation time is assumed to be between forty and one hundred days at maximum Hanford flux.

Curies in Shipment	2,500	10,000	20,000
No. of Slugs Used (8" Hanford slugs)	15	50	110
Milligrams of inactive barium	285-715	950-2400	2100-5250
Milligrams of inactive barium/1000 C.	114-286	95-240	105-262.

During Rala Run #28 the personnel of the Technical Division sampled the cell ventilation air and the gases from dissolver and other process vessels. The results of this investigation are contained in a memorandum from C. P. Coughlin and S. E. Beall to C. E. Winters, dated December 7, 1948; subject: "Radiation Hazard Measurements for the Period November 26 to December 3, 1948; C.F. No. 48-12-104.

As a result of this study, a filter unit, composed of two thicknesses of American Air Filter Company glass fiber F.G. #50 and one thickness of C.W.S. #6 paper are being installed in the cell ventilation system prior to the next run. Additional samples will be taken of the gases from the process vessels and dissolver to allow the design of equipment for their decontamination.

E J Witkowski
E. J. Witkowski

L B Emlet
L. B. Emlet

wp

[redacted]

~~SECRET~~ B-13

OAK RIDGE NATIONAL LABORATORY
CENTRAL FILES NUMBER
49-1-159

B-137

RUN 29

anal.

Date January 17, 1949

Subject Analytical Data Ba Run #29,

Shipment # 37

By E. I. Wyatt

To L. B. Emlet

File [illegible]

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1-17-49

To: L.B. Emlet
From: E.I. Wyatt

Analytical Data
Ba Run #29
L.S.T. 2020-1-14-49
Shipment #37

Classification cancelled ~~or Changed~~
TO
By Authority of
ATG AUG 27 1971
Name Title Date

OAK RIDGE NATIONAL LABORATORY
CENTRAL FILES NUMBER
49-1-159

Batch	Curies	Slugs	A-8	A-6	% Loss
A	2434	27.7	98	11	0.36
B	627	10.0	159	---	5.19
Total	3061	37.7	159	11	5.55

Other Cell A Losses

	Curies	% Loss
Wash of Ext. Cake (8WW)	14	0.46
Metathesis (8WC)	471	----
Metathesis Cake Wash (8WCW)	527	17.21
A-9 Rinse	37	1.21
Total Cell A Losses	748	24.43

Cell B Losses

Electrolysis (3WPb)	17	0.55
B-6 Rinse	26	0.85
Fuming Nitric Waste	25	0.82
HCl-Ether Waste	230	7.51
Total Cell B Loss	298	9.73

Total Losses

Product

Product in B-6 (Radiochemical)	2352	76.8%
Material Balance (Through B-6)	----	102.0%
Total Product shipped (Radiochemical)	----	-----
Total Product shipped (Skyshine)	1545 LA Units* (2163)*	50.5%
Material Balance through Skyshine		84.6%

CLASSIFICATION CANCELLED
ADD signature: A S Quist Date: 1/19/95
Single rereview of CCRP-declassified documents was authorized by DOE Office of Declassification memo of August 22, 1994.

cc:
E. Witkowski
Central Files

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OAK RIDGE NATIONAL LABORATORY
CENTRAL FILES NUMBER
49-1-73

B-137

re: Run 28

Date January 7, 1949

Subject Rala Production

By R. W. Cook

To N. E. Bradbury

File ____________

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L B Emlet

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David R Hamrin 2/6/95
Technical Information Officer Date
ORNL Site

TRANSMITTAL DATED 3-8-49

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1 3 C-c

UNITED STATES
ATOMIC ENERGY COMMISSION

In Reply Refer To Classification Cancelled [redacted]
RMRP:JS TO
By Authority of
ATG AUG 27 1971
Name Title Date

Oak Ridge, Tennessee
January 7, 1949

OAK RIDGE NATIONAL LABORATORY
CENTRAL FILES NUMBER
49-1-73

Dr. N. E. Bradbury
Director of Los Alamos Scientific Laboratory
Post Office Box 1663
Santa Fe, New Mexico

Subject: RALA PRODUCTION

Dear Dr. Bradbury:

Reference is made to your letter addressed to Mr. J. C. Franklin, dated December 21, 1948, above subject.

Answers to your questions are being prepared and will be forwarded to you as soon as they can be assembled. Some comments can be made at the present time regarding the points you have raised.

Hanford rather than Clinton slugs will be used as the source of radioactive barium. Source #28, which was shipped to you on November 22, 1948, was the first produced from Hanford material. Source #29 will probably leave Oak Ridge on January 14.

We do not plan to add any new production facilities in order to produce 10,000 and 20,000 curies sources. The Operating Division of the Oak Ridge National Laboratory believes that the larger souces can be produced with' present equipment providing, of course, that Hanford material is used. A better indication of the ability to produce the larger sources will be had following the next run.

A Hanford group, headed by Mr. W. M. Harty, recently made an extensive study of RaLa production at ORNL and, therefore, understands thoroughly the requirements of the process. In addition, this group was at the conferences on November 8 and 9, 1948, during which time discussions were held with the ORNL Operating Division and Drs. Mueller, White, and Mr. Leary of your Laboratory. Hanford is giving special attention to the slugs bring irradiated for the next run in order to furnish a maximum ratio of active to inactive barium.

Since no new facilities will be added for the larger sourses, it is probable that they can be supplied starting February 1, 1950. However, we were advised in September, 1948, by Mr. Walter J. Williams that the date for starting production of these sources is June 1, 1950.

CLASSIFICATION CANCELLED
JG Morgan 1-18-95
ADD signature Date
Single rereview of CCRP-declassified documents was authorized by DOE Office of Declassification memo of August 22, 1994.

S[redacted]

CA[redacted]

[redacted]

Dr. N. E. Bradbury
January 7, 1949

The question of the ratio of active to inactive barium is covered in an ORNL memorandum from E. J. Witkowski and L. B. Emlet to C. N. Rucker, No. CF 48-12-192, entitled "RaLa Production from Hanford Slugs and Product Contamination Studies", dated December 15, 1948. Copies 4 and 5 were sent to your Mr. Leary and Dr. Mueller, and should be received at Los Alamos within a few days.

Yours very truly,

R. W. Cook
Deputy Manager
Oak Ridge Operations

CC: C. L. Tyler, SFOO
A. H. Holland, AEC

Shilling:mw

COPY
Forwarded By
C. N. RUCKER

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2. C. N. Rucker
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B-137

No: 28-29

34

Date February 1, 1949

File

Subject Rala Production Problems

Those Eligible To Read The Attached

By C. N. Rucker

Copy # 9A

To A. H. Holland

J. L. Steakley

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4

9 11 A

OAK RIDGE NATIONAL LABORATORY

DIVISION OF

CARBIDE AND CARBON CHEMICALS CORPORATION

UCC

POST OFFICE BOX P
OAK RIDGE, TENNESSEE

CENTRAL FILES NUMBER
49-2-34

February 1, 1949

U. S. Atomic Energy Commission
Post Office Box E
Oak Ridge, Tennessee

Classification Cancelled
~~Or Changed To~~
By Authority Of DOC
By EEB Date AUG 27 1971

Attention: Dr. A. E. Holland, Jr.

Subject: RALA PRODUCTION PROBLEMS

CLASSIFICATION CANCELLED
ADD signature 1/18/95 Date
Single rereview of CCRP-declassified documents was authorized by DOE Office of Declassification memo of August 22, 1994.

Reference: (1) Letter to C. N. Rucker from A. E. Holland, dated December 29, 1948; subj: Rala Production Facilities; C.F. No. 48-12-294.
(2) Letter to C. N. Rucker from A. E. Holland, dated January 7, 1949; subj: Re: Size of Rala Sources; C.F. No. 49-1-72.
(3) Letter to N. E. Bradbury from E. V. Cook, dated January 7, 1949; subj: Rala Production; C.F. No. 49-1-73.

Gentlemen:

The second Rala run, using Hanford-irradiated slugs, was completed and shipped to Los Alamos on January 16, 1949. The results of these two runs are listed below and compared to the average of five batches made during 1948 using Oak Ridge slugs.

	4" HANFORD SLUGS				ORNL SLUGS	
	Run #8		Run #9		Avg. of 5 Runs	
*Curies in Dissolver	2619	100%	3061	100%	3520	100%
Metal Used	184 lbs.	--	152 lbs.	--	2295 lbs.	--
Slugs	36	--	30	--	893	--
Total Losses	1239	47.3%	1516	49.5%	1110	31.5%
Cell A	681	26.0%	748	24.4%	743	21.1%
Cell B	201	7.6%	298	9.7%	177	5.0%
Unexplained	357	13.7%	470	15.4%	192	5.4%
Curies Shipped	1380	52.7%	1545	50.5%	2410	68.5%

* All curies corrected to L.S.I.

[redacted]

[redacted]

[redacted]

You will notice that the chemical yields of the two runs using Hanford slugs are below the 1948 average of batches using ORNL slugs. These low yields, however, are not believed to be the result of using Hanford slugs but are caused by larger mechanical losses during the initial separation step (Cell A). Slightly higher losses also occurred in the final purification stages (Cell B) of the two runs using Hanford slugs. No reason is known for the high unexplained losses in the two Hanford runs, but it is possible that the more concentrated solutions handled may account for a portion of the material lost.

It was originally anticipated that a 4" Hanford slug would contain about 275 curies of Ba^{140} at time of discharge from the pile. The results of the dissolver solution analysis of the last two runs indicate that an average of 140 curies are contained in each slug or about half of the calculated amount. For this reason, plus the higher losses experienced, it is evident that process improvements and equipment modifications will be necessary before 10,000 or 20,000-curie batches can be produced at ORNL.

In preparation for increasing the batch size of RaLa runs from 2,000 curies to 10,000 and perhaps 20,000 curies, the problem of improving the present process was referred to our Technical Division. They can start an investigational program within a month or so aimed at improving the process equipment and operating facilities. It is believed that this program can be completed by October, 1949, which will allow several months for any equipment changes necessary before attempting the larger sized batches in February, 1950, as requested by Los Alamos.

It is our understanding, however, that the Atomic Energy Commission has directed the General Electric Company at Hanford to proceed with the plans outlined in Report HW-11657 (Feasibility of RaLa Production at Hanford). That is, to design and build a plant and have it completed by the middle of 1950 for the production of 10,000 and 20,000-curie batches. If this is the case, it does not appear desirable to divert any of the Laboratory's effort from other important projects to work on RaLa improvement. This would mean that ORNL would continue to supply 2,000-2,500-curie batches with no attempt at increasing the batch size until Hanford was ready to assume the production responsibilities. Your comments on this point are requested.

The following information is supplied in answer to the several questions listed in reference letter (3):

1. The use of Hanford slugs has many advantages over ORNL-irradiated material. It is expected that Hanford slugs will be used for all future RaLa runs at this Laboratory.

[redacted]

2. It is recommended that the exposure period for Hanford slugs be limited to a minimum of forty (40) days and a maximum of one hundred (100) days at maximum Hanford flux. Irradiation periods of less than forty (40) days will reduce the Ba^{140} content, while periods of greater than(100) one hundred days will increase the quantity of inactive barium. It is our understanding that a more limited control of the irradiation period will interfere with the Hanford plutonium production schedules.

Since the La^{140} is "milked" from the Ba^{140} before it is used at Los Alamos, it is not clear to us as to why the inactive barium content is a limiting factor. Information on this point will be appreciated.

3. The lower concentration of Ba^{140} in the Hanford uranium will result in the use of a larger number of slugs; the inactive barium content should remain essentially the same as previously estimated.* The revised estimate is as follows:

Curies in Shipment	2500 C.	10,000 C.	20,000 C.
(1) No. of Slugs (4" Hanford)	60	240	480
(2) mg of inactive barium	250 to 725	1000 to 2900	2000 to 5800
mg of inactive barium/1000 C.	100 to 290	100 to 290	100 to 290

(1) Based on yield of 42 C/slug at L.S.T.
(2) Calculated on 40 to 100-day exposure at maximum Hanford flux.

* Memo to C. E. Rucker from E. J. Witkowski and L. B. Emlet, dated December 15, 1948; subj: RaLa Production from Hanford Slugs and Product Contamination Studies; C.F. No. 48-12-192.

4. Providing it is decided that RaLa production will continue at ORNL and that a process improvement program is started immediately, we are reasonably certain that 10,000-curie RaLa batches can be produced by February, 1950.

5. It is believed that the modified facilities will be adequate to produce 20,000 curies of RaLa in one batch, although this may be more difficult than originally anticipated. The results of the contemplated development work will give a more definite answer to this problem.

[redacted]

6. We are not acquainted with the design of the container required for the Los Alamos facilities. It is reasonable to assume that modifications can be made in either ORNL or Los Alamos equipment to accommodate a carrier mutually agreed upon. It is suggested that a meeting of the interested parties be planned to determine the details of this carrier.

7. The product can be shipped as the nitrate instead of the chloride.

8. The present investigation of impurities in the product is being continued. Evidence from "dummy" runs indicates that the major source of the "organic" contaminate is from the Tygon tubing previously used. This Tygon has been replaced with fluorothene tubing and initial results appear promising. Unfortunately, the nickel contaminate from the Hastelloy transfer vessel used in Run #29 prevented an evaluation of the product for the organic impurities. There is evidence that Los Alamos may introduce some organic impurities caused by the beta-gamma deterioration of the oxalate during the La^{140} "milking" operation. It is suggested that Los Alamos investigate this phase of the problem.

Reference letter (1) requests information concerning the atmospheric contamination during RaLa Run #29. The Technical Division is preparing a complete report on the results of this study. In general, approximately three (3) curies of activity passed through the filters in the various off-gas and vent lines and into the atmosphere during the seven (7) days of operation or an average of about 15 mc/hour. There were peaks in the output that reached a rate of 300 mc/hour. The study of equipment improvement, scheduled by the Technical Division, will investigate this phase of the problem further.

Very truly yours,

OAK RIDGE NATIONAL LABORATORY

Original Signed By
C. N. Rucker

C. N. Rucker
Executive Director

[illegible]

1-3. A. H. Holland
4. C. E. Center
5. L. B. Emlet
6. A. M. Weinberg
7. M. D. Peterson
8. E. J. Witkowski
9. F. L. Steahly
10-11. C. N. Rucker

[redacted]

173

Routine Prob.

~~S-E-C-R-E-T~~

This document consists of 12 pages.
No. 7 of 11 copies.
Series A.

OAK RIDGE NATIONAL LABORATORY
CENTRAL FILES NUMBER
49-4-35

Date: April 4, 1949

Subject: Air Activity During RaLa Runs #30 and 30A

By: H.J. McAlduff and J.S. Cheka

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4-5-49

Air Activity During RaLa Runs #30 and 30A
(2-14 to 2-27-49)

by

H.J. McAlduff and J.S. Cheka
Health Physics Division
Oak Ridge National Laboratory

The comparison of air sampling data obtained during the RaLa run of January 10-16, 1949 (cell ventilation air filtered) with those obtained during previous runs (cell ventilation air unfiltered) showed a great improvement in particle incidence and general area contamination, as a result of the filter installation. Air monitoring instruments in the immediate vicinity of, and inside, 706-C and D buildings, however, gave evidence that significant amounts of airborne contamination were still being produced by the RaLa operation, the largest amount of which was believed to be coming from inside 706-D. Because of this it was decided to monitor the next run, with greater attention being paid to the amount of contamination being produced inside the building, and to determine if possible what specific operations were the greatest contributors.

The complexity of RaLa operations is such that seldom, if ever, are two runs exactly similar. The run of February 14 to February 27, 1949 was no exception. Operating difficulties were experienced which necessitated a double run, therefore conditions existing during this run are considered extreme rather than representative.

Air activity in 706-D and 706-C commenced rising noticeably on 2-16, two days after the start of RaLa run #30, remaining higher than normal until the completion of Run #30A on 2-27-49. The highest periods of activity obtained on 2-18, 2-22, 2-26 and 2-27 when it became necessary to evacuate the buildings for periods of from 10-35 minutes. The air activity associated with RaLa operations during these runs may be considered not primarily a particle problem although the difference seems to be a fine point of definition of a particle. Radio-autographs of filters from constant air monitors within 706-D and 706-C buildings, during the runs, gave a pattern of diffuse activity over the entire surface of the filter paper, as differentiated from spots of localized activity which are recorded as separate particles. Localized spots of activity were in evidence on these filters, but in some instances the density due to the diffuse activity was such as to make the detection of less intense localized areas impossible. Such diffuse activity has been noticed in the past and had been associated with chemical operations. The physical form of the activity is unknown, but thought possibly to be due to fumes, or vapor.

The data comprising results obtained from radio-autographing constant air monitor filters from inside 706-D and 706-C buildings are presented in Tables I and II respectively. All filters were exposed to film for 24 hours, and in some instances counts were obtained, using a standard $\beta-\gamma$ counter, prior to filming. Where counts were obtained the value in μc/sample is given at the time of counting plus the value of μc/cc of air based on an air flow of 300 ft /hour. Total particle counts are listed, being the number of spots of localized activity discernible on the radio-autograph of the filter.

Table III gives the results of radio-autographing filters from a constant air monitor sampling cell ventilation air from 706-D stack. Sampling of the stack effluent was not started until 2-25, and then to determine if the activity in 706-D was coming from the stack. Negative indications obtained by this method were further substantiated by sampling done by the Technical Division, whose samples showed no greater amount of activity passing through the cell ventilation air filter than had been experienced in the January RaLa run.

Table IV lists various operations in progress during each day of the runs. No elaborate effort was made to break the operations down more accurately, as the operating difficulties experienced would not make such a breakdown any more valuable in the interpretation of results.

Table V lists the results obtained from radio-autographing filters from 3 outside constant air monitors bracketing 706-D building. Some diffuse activity was evident on these filters, indicating a moderate amount of dispersal of contamination about the area.

Decay curves were run on two of the filtron samples taken during this run. One was a 42 hr. sample taken on the East loading platform of 706-D taken off at 0835 2-23. The other was a 30 minute sample taken immediately thereafter on the North Side of 706-D. A graphical analysis of the decay curves indicated an 8 day fraction, a 22 hr. fraction and some shorter-lived material. As would be expected, the short sample had the smaller percentage of the longer-lived activity. Any longer-lived fraction that may have been present was not distinguishable in a 14 day decay period.

SECRET

Table I
Constant Air Monitors (Inside 706-D)

2nd Level (NW)				2nd Level (SE)					3rd Level (Center)				
ection Period (hrs)	Particles	μc/sample x 10^{-2}	μc/cc x 10^{-12}	Collection Time	Period (hrs)	Particles	μc/sample x 10^{-2}	μc/cc x 10^{-12}	Collection Time	Period (hrs)	Particles	μc/sample x 10^{-2}	μc/cc x 10^{-12}
.20	110												
.46	6								~0800	—	0		
.48	42	2.0	49	0520	~18	8	0.5~	~32.6	0820	~48	58	0.9	~22.1
21.	33	4.1	230	0800	26.5	12	2.9	129.	0430	23	6	4.0	205
2.5	10	3.3	1710										
25	8	>9.8	> 462	0900	25	30	2.9	126	0900	28.5	26	3.9	161
3.5	36			1200	3	5			1205	3	30		
21	34			0845	21	42			0845	21	58		
24	143			0835	24	18			0830	24	18		
24	61	>9.8	>481	0815	24	15	0.6	29.4	0815	24	50	2.4	118
24	290	>9.8	>481	0815	24	116	3.5	172	0815	24	128	8.1	395
19	1	8.8	431	0300	19	24	5.7	279	0305	19	36	6.8	332
1	4	>9.8	>11500	0830	6.5	32			0405	1	12	6.1	7180
7.5	20			1130	3	36			0525	1.3	28	5.7	5060
8.5	8								1130	6	49		
									2000	8.5	10		
5.5	—			0820	21	3			0130	5.5	3		
5.3	2								0825	7	2		
1.75	4								1115	~3	14		
2.5	18												
1.3	30			0810	24	44			0815	21	26		
7	36	>9.8	>680	0115	17	39	3.4	237	0115	17	30	4.2	292
0.6	—	>9.8	>1090						1145	10	70	>9.8	>1153
4	58			0145	14.5	36	>9.8	>797	0145	14	57		
7.25	19	**		0900	7.25	60			0900	7.25	80		
1.5	—	>9.8	>7720	1030	1.5	0	>9.8	>7720	1100	2	38	>9.8	>5760
	59			0830		10			0845		77		
									2210		136		

acuated ** 8 mr - Victoreen 263

ove values of μc/cc were calculated using 300 ft3/hr as the air flow for the constant air monitors. Recent measure- indicate this air flow value to be inaccurate, consequently values as noted show a greater divergence between stations ctually exists.

SECRET

Table II
Constant Air Monitors (Inside 706-C)

ate	1st Level (W) Collection Time	Period (hrs)	Particles	μc/sample x 10^{-2}	μc/cc x 10^{-12}	2nd Level (S) Collection Time	Period (hrs)	Particles	μc/sample x 10^{-2}	μc/cc x 10^{-12}	2nd Level (N) Collection Time	Period (hrs)	Particles	μc/sample x 10^{-2}	μc/cc x 10^{-12}
-14	~0800	~48	1			~0800	~48	2			~0800	~48	83		
-16	0800	48	5	0.2	4.6	0800	48	6	0.07	1.8	0800	48	60	0.63	15.2
-17	0800	24	11	0.31	15.2	0800	24	2	0.013	0.64	0800	24	7	3.3	162
-18	0800	24	27	3.3	162	1230	~28.5	65	0.95	280	0800	24	74	>9.8	>480
											~1230	~4	10		
-21	1035	~74	235	1.9	30	1045	~70	77	.97	16	1040	~70	206	3.5	59
											1415	3.5	176		
-22	0800	21.5	140	1.2	65	0800	21.5	123	1.43	79	0800	19	171	7.5	466
-23	0800	24	15			0800	24	36			0800	24	12		
	2100	13	8												
-24	0800	11	0			0800	24	0			0800	24	0		
-25	0800	24	10			0800	24	25			0800	24	22		
	-----	—	12												
-26						1930	35.5	45	>9.8	>325					
-28						0800	36.5	60			0800	72	--	>9.8	>161
-1						0800	24	14			0800	24	13		

Note: The above values of μc/cc were calculated using 300 ft^3/hr as the air flow for the constant air monitors. The divergence in values noted between stations is subject to verification pending new air flow measurements.

Table III
706-D Stack Samples

Date		Time	Particles	C/M	Remarks
2-25	(1)	1130 - 1330		1497	
	(2)	1330 - 1430		600	
	(3)	1430 - 1530		736	
	(4)	1530 - 1730	7		
	(5)	1730 - 1930	23		
	(6)	1930 - 2130	58		
	(7)	2130 - 2330	85		
2-26	(8)	2330 - 0130	202		
	(9)	0130 - 0330	262		
	(10)	0330 - 0530	211		
	(11)	0530 - 1130	56		
	(12)	1130 - 1330	111		
	(13)	1330 - 1530	199		
	(14)	1530 - 1730	302		
	(15)	1730 - 1930	114		
	(16)	1930 - 2130	13		* large amount
	(17)	2130 - 2330	95		diffuse activity
2-27	(18)	2330 - 0130	86		
	(19)	0130 - 0530	66		
	(20)	0530 - 0730	183		
	(21)	0730 - 0930	94		
	(22)	0930 - 1130	58		* hot spots
	(23)	1130 - 1330	104		
	(24)	1330 - 1530	268		* hot spots
	(25)	1530 - 1730	191		
	(26)	1730 - 1930	167		
	(27)	1930 - 2130	169		
	(28)	2130 - 2330	95		
2-28	(29)	2330 - 0130	85		
	(30)	0130 - 0330	79		
	(31)	0330 - 0530	76		
	(32)	0530 - 0730	56		
	(33)	0730 - 1030	46		
	(34)	1030 -			
3-1	(34)	1300	52		* Diffuse activity

Table IV
706-D Operations (RaLa Run)
2-14 to 2-28-49

2-14 2-15 2-16 2-17	Loading slugs, coating removal, transfer operations, extraction, settling and waste neutralization.
2-18	Extraction cake washes 0320 - 1025 Metastasis - (1020 - 2015) Metastasis washes 2000 to 0200 (2-19)
2-19	(0120) Transfer to Cell B Electrolysis (0150 to 1150) 6 ρ sample at 1300 B-6 volume reduction (1300 - 1715) In glassware 1700 to 1230 (2-20) Various recovery operations in progress from 1400 to 2330
2-20	Product dried (1300 - 1700) Pb removal 2100 - 0045 (2-21)
2-21 2-22 2-23 2-24	Loading, coating removal, dissolving, transfers, waste neutralization, metastasis washes, metastasis.
2-25	Transfer to Cell B (2240) Electrolysis 2130
2-26	Electrolysis ended (0740) Sample 6 ρ (very high air activity - evacuation) 0935-1230 (B-6 volume reduction) Hot samples taken Pb removal
2-27	0400 Out of glassware 1615 - 2015 Product evaporation and drying Product in Cell B 2015 (2-27 to 0900 2-28) Product shipped 100 2-28

Air Activity During RaLa Runs #30 and 30A

Table V
Outside Constant Air Monitors

Date	#1 125' East 706-D Total Particles	#2 200' NNW 706-D Total Particles	#5 600' SW 706-D Total Particles
2-14	3	1	0
2-18	20	7	
2-21	4	12	53
2-22		4	
2-23	13	4	34
2-24		0	
2-28	10	23	16
3-7	4	5	2

Summary

Due to the variable factors inherent in RaLa operations the data presented are of qualitative significance only. However, an examination of these data allows for the following generalizations to be made.

(1) Air activity does not seem to be confined to any one particular operation in the process.

(2) Highest air activity seems to come in sudden increases, although a gradual rise frequently occurs before the increase.

(3) A moderate amount of activity is still passing through the cell ventilation air filter, however, the magnitude of the activity is less than that being detected inside the building.

(4) Some phases of the operation when the product is in glassware contribute to high activity of the cell ventilation air.

(5) Taking of "Skyshine" readings and the removal of the product carrier from Cell B seems to result in the dispersal of particles of very high specific activity, especially

on the 3rd level. During this period similar particles were detected in the filtered cell ventilation air.

(6) No distinguishable 300 hr. activity was present during the period of highest air activity in the course of the run.

H. J. McAlduff
H. J. McAlduff

J. S. Cheka
J.S. Cheka

HJMcA/JSC:rr

Data on Runs

OAK RIDGE NATIONAL LABORATORY
CENTRAL FILES NUMBER
49-3- 1

~~SECRET~~

B-137

RUNS 30 30A To CF

~~Mat. General "B"~~

3-1-49

Date February 28, 1949

Subject Analytical Data Ba Run 30-30A, Shipment #38

By E. I. Wyatt

To L. B. Emlet

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Analytical Data
Ba Run 30-30A
LST 1330 2-27-49
Shipment #38

2-28-49

CENTRAL FILES NUMBER

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Batch	Curies	Slugs	A-8	A-6	% Loss
(30)A	682	63.2		17	0.41
B	713	60.3		38	0.93
C	631	50.1		12	0.29
D	251	56.2		26	0.63
E	224	59.7		15	0.37
F	131	40.4	39		0.95
Heels	1	0.5			---
(30A)A	362	68.9		28	0.69
B	297	60.9		31	0.76
C	269	58.0		25	0.61
D	266	57.5		32	0.78
E	224	53.5	62		1.51
F					
Heels	46	10.5			
Total	4097	639.7	101	224	7.93%

Other Cell A Losses

6WM Recovery (Run 30)	6	0.15
Wash of Ext. Cake Run 30(6WW-1)	3	0.07
Al Heels (Run 30)	1	0.02
Al Heels (Run 30A)	46	1.12
Wash of Ext. Cake(Run 30A)(8WW)	31	0.76
Metathesis (Run 30A) (8WC)	307	----
Metathesis Cake Wash (Run 30A(8WCW)	204	4.98
A-9 Rinse	52	1.27
Total Cell A Losses	668	16.30%

Cell B Losses

	Curies	
HCl Ether Waste (Run 30)	64	1.56
B6 Waste #2 (Run 30)	216	5.27
B6 Waste #3 (Run 30)	11	0.27
Fuming Nitric Waste (30A)(3WFN)	61	1.49
Electrolysis (30A)(3WPb)	79	1.93
B6 Rinse (30A)	19	0.46
HCl Ether Waste (30A)	645	15.74
Total Cell B Losses	1095	26.72

Product

Product in B-6 (Radiochemical)	2300	56.1%
Material Balance thru B-6		81.5%
Total Product shipped (Radiochemical)	1575	~~25.5%~~ 38.4% - changed per Wyatt. 3-2-49 wp.
Total Product shipped (Skyshine)	1345	32.8%
Material Balance thru Skyshine		75.9%

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Anal. Data on Runs

OAK RIDGE NATIONAL LABORATORY

CENTRAL FILES NUMBER

49-3-255

[redacted]

RUN 31

B-137

INV. 64

To CF

Date March 25, 1949

File

Subject Analytical Data, Run #31, Shipment #39

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By E. I. Wyatt

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From: E.I. Wyatt

Analytical Data
Ba Run #31
L.S.T. 0645-3-24-49
Shipment #39

OAK RIDGE NATIONAL LABORATORY
CENTRAL FILES NUMBER
49-3-255

Batch	Curies	Slugs	A-8	A-6	% Loss
A	3048	35.8	2005	93	1.49%
B	2946	35.6	509	27	8.14%
Al Heels	258	3.9	---	--	-----
Total	6252	75.3	509	93	9.63%

	Curies	% Loss
Other Cell A Losses		
Wash of Ext. Cake (8WW)	225	3.60
Metathesis (8WC)	381	----
Metathhesis Cake Wash (8WCW)	389	6.22
A-9 Rinse	20	0.32
A-1 Heels	258	4.13
Total Cell A Losses	1494	23.90%
Cell B Losses		
Electrolysis	26	0.41
B6 Rinse	25	0.41
Fuming Nitric Waste	80	1.28
HCl-Ether Waste	299	4.78
Total Cell B Losses	430	6.88%
Total Losses	1924	30.78%
Product		
Product in B6 (Radiochemical	3570	57.10%
Material Balance (through B6)		81.4%
Total Product shipped (RC)	3166	50.6%
Total Product shipped (Skyshine)	3244	51.9%
Material Balance (through Skyshine)		82.7%

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By
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B-137

RUN 32

Date April 22, 1949

Subject Analytical Data Ba Run #32, Shipment #40

By E. I. Wyatt

To L. B. Emlet

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Analytical Data
Ba Run #32
L.S.T. 1700-4-21-49
Shipment #40

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Batch	Curies	Slugs	A-8	A-6	% Loss
A	3220	36.8	506	34	.57%
B	2546	32.9	438	152	7.31%
A-1 Heels	223	6.0			
	5989	75.7	438	34	7.88%

Other Cell A Losses

	Curies	% Loss
Wash of Ext. Cake (SWW)	154	2.57
Metathesis (SWC)	242	
Metathesis Cake Wash (SWCW)	540	9.02
A-9 Rinse	21	0.35
A-1 Heels	223	3.72
Total Cell A Losses	1410	23.54%

Cell B Losses

	Curies	% Loss
Electrolysis	27	0.45
B6 Rinse	11	0.18
Fuming Nitric Waste	66	1.10
HCl-Ether Waste	406	6.78
Total Cell B Losses	510	8.51%

	Curies	% Loss
Total Losses	1920	32.05%

Product

	Curies	%
Product in B6 (Radiochemical)	4240	70.8%
Material Balance (through B6)	----	94.8%
Total Product Shipped (RC)	3757	62.7%
Total Product shipped (Skyshine)	3800	63.4%
Material Balance (Through Skyshine)		95.5%

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RUN 33

OAK RIDGE NATIONAL LABORATORY
CENTRAL FILES NUMBER
49-6-8

Date May 28, 1949

Subject Analytical Data, Run #33, Shipment #41

By E. I. Wyatt

To L. B. Emlet

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Analytical Data
Ba Run #33
L.S.T. 2200-5-27-49
Shipment #41

5-28-49

Batch	Curies	Slugs	A-8	A-6	% LOSS
A	2813	40.8	114	28	0.52
B	2270	33.0	318	43	5.94
A-1 Heels	275	4.6	----	---	----
Total	5358	78.4	318	28	6.46

Other Cell A Losses	Curies	% Loss
Wash of Ext Cake (8WW)	83	1.55
Metathesis (8WC)	316	----
Metathesis Cake Wash (8WCW)	656	12.24
A-9 Rinse	18	0.34
A-1 Heels	275	5.13
Total Cell A Losses	1378	25.72
Cell B Losses		
Electrolysis	29	0.54
B6 Rinse	260	4.85
Fuming Nitric Waste	70	1.31
HCl-Ether Waste	260	4.85
Total Cell B Losses	619	11.55
Total Losses	1997	37.3%
Product		
Product in B6 (Radiochemical)	3795	70.8%
Material Balance (Thru B6)	-----	97.1%
Total Product shipped (RC)	3205	------
Total Product shipped (Skyshine)	3360	62.7%
Material Balance (thru Skyshine)	----	100.0%

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RUN 34

49-7-140

Date July 15, 1949

Subject Analytical Ba Run # 34 - Shipment # 42

By E. I. Wyatt

To L. B. Emlet

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Analytical Data
Ba Run #34
LST 1215-7-14-49
Shipment #42

Batch	Curies	Slugs	A-8	A-6	% Loss
A	3157	34.6	157	21	0.36
B	2370	32.0	211	132	3.57
Al-Heels	376	6.25	---	---	----
	5903	72.85	211	21	3.93

Other Cell A Losses	Curies	% Loss
Wash of Ext. Cake (8WW)	139	2.35
Metathesis (8WC)	274	----
Metathesis Cake Wash (8WCW)	515	8.72
A-9 Rinse	21	0.36
A-1 Heels	376	6.37
Total Cell A Losses	1283	21.73

Cell B Losses		
Electrolysis	28	0.47
B6 Rinse	10	0.17
Fuming Nitric Waste	33	0.56
HCl-Ether Waste	370	6.27
Total Cell B Losses	441	7.47

Total Losses	1724	29.21%
Product		
Product in B6 (Radiochemical)	4145	70.22%
Material Balance (Through B6)		92.43%
Total Product Shipped (RC)	3732	------
Total Product Shipped (Skyshine)	4284	72.57%
Material Balance (Thru Skyshine)	-----	101.77%

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RUN 35

49-8-258

Date August 26, 1949

Subject Analytical Data Run #35, Shipment #43

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Analytical Data
Ba Run #35
L.B.T. 1515-8-24-49
Shipment #43

8-26-49

258

Batch	Curies	Slugs	A-8	A-6	% Loss
A	2195	35.9	217	23	0.58
B	1644	35.1	88	21	0.53
A-1 Heels	109	1.5	--	--	----
	3948	72.5		44	1.11

Other Cell A Losses	Curies	% Loss
6 WM Recovery	92	2.33
6WM Recovery #2	69	1.75
Wash of Ext. Cake (8WW)	653	16.54
Metathesis (8WC)	110	-----
Metathesis Cake Wash (8WCW)	121	3.07
A-9 Rinse	25	0.63
A-1 Heels	109	2.76
Total Cell A Losses	1113	28.19%

Cell B Losses	Curies	% Loss
Electrolysis	70	1.77
B6 Rinse (First)	35	0.89
B6 Rinse (Second)	18	0.45
Fuming Nitric Waste	37	0.94
HCl-Ether Waste	146	3.70
Total Cell B Losses	306	7.75

	Curies	% Loss
Total Losses --------------------------	1419	35.94
Product		
Product in B6 (Radiochemical)	1536	38.91
Material Balance (Through B6)	----	68.87
Total Product shipped (Radiochemical)	1300	
Total Product shipped (Skyshine)	1750	44.33
Material Balance (Thru skyshine)	----	80.27

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RUN 36

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CENTRAL FILES NUMBER
49-10-87

Date October 14, 1949

Subject Ba Run #36, Shipment #44

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Analytical Data
Ba Run #36
L.S.T. 1545-10-13-49
Shipment #44

10-14-49

87

Batch	Curies	Slugs	A-8	A-6	% Loss
A	3363	40.7	148	55	0.93
B	2255	30.7	260	57	4.41
A-1 Heels	275	4.0	---	--	----
Total	5893	75.4	260	55	5.34

Other Cell A Losses	Curies	% Loss
Wash of Ext Cake (8WW)	18	0.305
Metathesis (8WC)	274	
Metathesis Cake Wash (8WCW)	406	6.89
A-9 Rinse	29	0.49
A-1 Heels	275	4.67
Total	1043	17.70

Cell B Losses		
Electrolysis (3WPB)	34	0.58
B-6 Rinse	19	0.32
Fuming Nitric Waste (3WFN)	73	1.24
HCl-Ether Waste	400	6.79
Total Cell B Losses	526	8.93

Total Losses	1569	26.63%
Product in B6 (Radiochemical)	4043	68.60%
Material Balance (Thru B6)	----	86.88%
Total Product shipped (Radiochemical)	3551	------
Total Product shipped (Skyshine)	4275	72.54%
Material Balance (Through Skyshine)	----	99.17%

cc: E.J. Witkowski
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RUN 37

Date November 25, 1949

Subject Analytical Data Ba Run #37, Shipment #45.

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Analytical Data
Ba Run #37
L.S.T. 2325-11-24-49
Shipment #45

11-25-49

Batch	Curies	Slugs	A-8	A-6	% Loss
A	3088	34.8	174	41	0.69
B	2364	30.9	278	50	4.69
A-1 Heels	478	6.2	---	--	----
Total	5930	71.9	278	41	5.38

Other Cell A Losses	Curies	% Loss
Wash of Ext Cake (8WW)	74	1.25
Metathesis (8WC)	433	----
Metathesis Cake Wash (8WCW)	525	8.85
A-9 Rinse	22	0.37
A-1 Heels	478	8.06
Total Cell A Losses	1418	23.91

Cell B Losses		
Electrolysis (3WPb)	31	0.52
B-6 Rinse	34	0.57
Fuming Nitric (3WFN)	100	1.69
Hcl-Ether Waste	248	4.18
Total Cell B Losses	413	6.96

Total Losses	1831	30.9
Product in B-6 (Radiochemical)	4226	71.3
Material Balance (through B-6)	----	95.7
Total Product Shipped (Radiochemical)	3844	----
Total Product Shipped (Skyshine)	4240	71.5
Material Balance (through Skyshine)	----	102.3

cc: E. J. Witkowski
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FILE RECORD SHEET

DOCUMENT NUMBER: 49-11-233

DATE: 11-25-49

SUBJECT: Analytical Data Ba Run # 37, Shipment # 45.

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Analytical Data
Ba Run #38
LST 1915 1-5-1950
Shipment #46

1-6-50

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Batch	Curies	Slugs	A-8	A-6	% Loss
A	3216	37	225	44	0.73
B	2392	31	234	57	3.89
A-1 Heels	405	6.8	---	--	---
	6013	74.8	234	44	4.62

Other Cell A Losses	Curies	% Loss
Wash of Ext. Cake (SWW)	24	0.40
Metathesis (SMC)	928	----
Metathesis Cake Wash (SMCW)	944	15.70
A-9 Rinse	70	1.16
A-1 Heels	405	6.74
Total Cell A Losses	1721	28.62

Cell B Losses	Curies	% Loss
Electrolysis (SWPb)	34	0.57
B6-Rinse	34	0.57
Fuming Nitric Waste(SWFN)	152	2.53
HCl Ether Waste	360	5.98
Total Cell B Losses	580	9.65

	Curies	% Loss
Total Losses	2301	38.27
Product in B6 (Radiochemical)	4612	76.7
Material Balance (thru B6)	----	106.0
Total Product shipped (Radiochemical)	4066	-----
Total Product shipped (Skyshine)	4670	77.7
Material Balance (Thru Skyshine)	----	116.0

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RUN 40

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50-3-77

Date March 17, 1950

Subject Analytical Data Ba Run #40 - Shipment #47.

By E. I. Wyatt

To L. B. Emlet

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3-17-50

To: L.B. Welet
From: T.I. Wyatt

Analytical Data
Ba Run #40
L.S.T. 1210 3-17-50
Shipment #47

4 4 A

Batch	Curies	Slugs	A-8	A-6	% Loss
A	3021	39	160	106	1.8
B	2402	35	485	59	8.39
A-1 Heels	361	5	----	----	----
	5784	79*	485	106	10.19

Other Cell A Losses

	Curies	% Loss
Wash of Ext. Cake (8EW)	27	0.47
Metathesis (8WC)	596	-----
Metathesis Cake Wash (8WCW)	579	10.01
A-9 Rinse	38	.66
A-1 Heels	361	6.24
Total Cell A Losses	1596	27.6

Cell B Losses

	Curies	% Loss
Electrolysis (3WPb)	32	.55
B6 Rinse	23	.40
Fuming Nitric Waste (3WFN)	78	1.35
HCl-Ether Waste	257	4.44
Total Cell B Losses	390	6.74
Total Losses	1986	34.3

	Curies	% Loss
Product in B6 (Radiochemical)	3506	60.6
Material Balance (thru B6)	---	88.8
Total Product Shipped (Radiochemical)	3148	----
Total Product Shipped (Skyshine)	3440	59.5
Material Balance (Thru Skyshine)	----	93.8

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RUN 41

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Date April 14, 1950

Subject Analytical Data Ba Run #41-
Shipment #48.

By E. I. Wyatt

To L. B. Emlet

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To: L. B. Emlet
From: E. I. Wyatt

Analytical Data
Ba Run #41
L.S.T. 0515 4-13-50
Shipment #48

4-14-50

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Batch	Curies	Slugs	A-8	A-6	% Loss
A	3349	33.9	373	25	0.387
B	2626	31.0	331	72	5.130
A-1 Heels	481	6.5	---	--	-----
	6456	71.4	331	25	5.52

Other Cell A Losses	Curies	% Loss
Wash of Ext. Cake (8WW)	35	0.54
Metathesis (8WC)	613	--
Metathesis Cake Wash (8WCW)	512	7.93
A-9 Rinse	20	0.31
A-1 Heels	481	7.45
Total Cell A Losses	1404	21.75

Cell B Losses	Curies	% Loss
Electrolysis (3WPb)	29	0.45
B6 Rinse	28	0.43
Fuming Nitric Waste (3WFN)	45	0.70
Hcl-Ether Waste	247	3.83
Total Cell B Losses	349	5.41

Total Losses	1753	27.16

	Curies	% Loss
Product in B6 (Radiochemical)	4456	69.0
Material Balance (thru B6)	----	91.2
Total Product Shipped (Radiochemical)	4136	----
Total Product Shipped (Skyshine)	4800	74.3
Material Balance (thru Skyshine)	----	101.5

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50-6-98

Date June 19, 1950

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Subject Analytical Data Barium Run #42

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To: L.B. Emlet
From: E.I. Wyatt

Analytical Data
Barium Run #42
L.S.T. 0630-6-16-50
Shipment #49

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Batch	Curies	Slugs	A-8	A-6	% Loss
A	2017	36.6	215	21	0.361
B	2437	38.0	194	54	0.929
C	1342	20.5	481	65	8.276
A-1 Heels	16	1.2	--	---	-----
	5812	96.3	481	75	9.57

Other Cell A Losses	Curies	% Loss
Wash of Ext. Cake (SWW)	53	0.91
Metathesis (SWC)	618	----
Metathesis Cake Wash (SWCW)	899	15.47
A-9 Rinse	20	.34
A-1 Heels	16	.28
Total Cell A Losses	1544	26.57

Cell B Losses	Curies	% Loss
Electrolysis (SWPb)	25	.43
B-6 Rinse	198	3.41
Total Cell B Losses	223	3.84

Total Losses	1767	30.41
Product in B6 Radiochemical	4033	69.4%
Material Balance (thru B6)	-----	96.4%
Total Product Shipped (Skyshine)	4140	71.2%

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RUN 44

INV. 64

Date May 21, 1951

Subject Analytical Data - Ba Run #44

By E. I. Wyatt

To M. E. Ramsey

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Analytical Data
Ba Run #44
LST 1322 5-20-51
Shipment #51

5-21-51

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To: M.E. Ramsey
From: E.I. Wyatt

Batch	Curies	Slugs	A-8	% Loss
A	8023	38.1	23	0.18
B	4817	29.5	28	0.21
A-1 Heels	291	1.9	--	----
	13131	69.5	51	0.39

Other Cell A Losses	Curies	% Loss
Metathesis Losses (1st Run)	378	----
Loss after 75% Recovery	84	0.64
1st Recovery metathesis Waste	390	2.97
2nd Recovery Metathesis Waste	116	0.88
A9 Rinse (Recovered)	337	----
A9 Rinse (Recovery #1)	5949	----
A9 Rinse (Recovery #2)	1354	10.31
8WM (Recovery #1)	1216	9.26
8WM Heels	844	6.43
8WM (Recovery #2)	250	1.90
Total Cell A Losses	4305	32.78

Extraction Cell Losses		
MWFE (Orig. Run)	25	0.19
ITNaVE (Orig. Run recovered)	53	----
MWHClE (after broken valve, recovered)	6677	----
ITNaVE (after broken valve, recovered)	1834	----
PE #1 (after broken valve, recovered)	21	----
MWFE (Recovery #1)	151	1.15
ITNaVE (Recovery #1)	1	0.01
MWHClE (Recovery #1)	77	0.59
Feed Tank Rinse (Recovery #1)	382	2.91
Feed Tank Rinse (Recovery #2)	20	0.15
MWFE (Recovery #2)	1114	8.48
ITNaVE (Recovery #2)	41	0.31
MWHClE (Recovery #2)	11	0.08
MWFN	25	0.19
PE-Rinse	22	0.17
Total Ext. Cell Losses	1869	14.23

Total Losses Accounted for		
Product		
PE-1 Recovery #1 plus #2	3204	24.4%
Total product shipped (Radiochemical)	3157	24.0%
Material Balance	9331	[redacted]1.06%

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DATE: June 13, 1951.

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No. 1 of 10
Series A

SUBJECT: 706-D Modification Project
First Full Level Production Run

TO: F.L. Steahly

COPY NO. 1A

FROM: R.H. Vaughan

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TO: F.L. Steahly

FROM: R.H. Vaughan

SUBJECT: 706-D Modification Project
First Full Level Production Run

1.0 Summary.

73 W slugs were dissolved and processed thru the new equipment, crud filters, process filters, and ion-exchange purification cubicles. The mechanical failure of a distribution valve in the ion-exchange cubicle spilled the ex- tected product yield, 12,000 curies, into the waste. The waste was subsequently reprocessed in stand-by equipment where 3000 curies was recovered as product and shipped 21 May 1951.

1.1 The slugs were charged to the dissolver on 13 May 1951, dissolved, processed, and the product shipped on 21 May 1951.

The slugs were dissolved in two batches and a heels dissolving, yielding the following amount of product.

CODE	C's Ba	C's Ba REFERRED TO LST	SLUGS APPROX.	C's/SLUG
1st METAL DISSOLVING	9066	8023	38.1	287
2nd METAL DISSOLVING	5443	4817	29.5	216.6
HEELS DISSOLVING	329	291	1.9	170
TOTALS	14,838	13,131	69.5	214 av.

All new equipment installed in conjunction with "A" cell operations functioned very satisfactorily. The crud filtration was fast - 2.8 gallons per minute average. The process filtration was also fast - 1.5 gallons per minute average. The losses thru these steps were very small, approximately 3%.

[redacted]

[redacted]

The transfer to the No.200 ion-exchange resin cubicle was made easily. The sampler unit worked very well and the feed solution pH adjustments were made with a minimum of effort. Adjustment of the feed flow to the column was handled very nicely thru the induction flowmeter. All multiway multiport plastic plug valves were operated with ease. This was prior to the time the product was to be stripped from the column and passed to the tantalum-lined product evaporator. Process valve - PV23 - was positioned so that the flow should pass to the evaporator. However, when the product was eluted from the column it passed into the main waste tank instead. This was discovered from the fact that the liquid level in this tank rose and with no apparent reason. The position indicator was changed so that the flow should be directed to other tanks but still the flow went only to the main waste tank. At this point a recovery run was instigated and all waste solutions in the resin cubicle were returned to "A" cell where they were combined with the original metathesis losses and dissolver heels. This combined solution was then processed starting with the extraction step as recovery run No.1. The ion-exchange purification was made thru stand-by equipment, the No.300 resin cubicle, identical with the No.200 cubicle and installed for just this purpose.

After the transfer of product from A-9 to the No.300 cubicle was made, a sample of the A-9 rinse revealed that approximately 50% of the product was still in A-9. This was believed due to an incomplete metathesis since the A-9 rinse sample had a precipitate in it. This A-9 rinse was in turn processed as recovery run No.2 starting with extraction step again.

[redacted]

Recovery run No.1 was carried thru up to the fuming nitric precipitation step. This product yield was retained in the product evaporator until recovery run No. 2 was completed up to the same point. The product yields were then combined in the product evaporator and processed to final completion as one run. The fuming nitric acid precipitation operation was carried out very satisfactorily. The transfer to the shipping cone was made easily and with only a small heel loss. The cone manipulator worked very well. The A-16 vacuum did not furnish enough air sweep thru the charging head for evaporation so the emergency jet from the charging heads to A-16 was utilized for this operation. This evaporation to dryness required 12 hours. 3157 curies of product was shipped to Los Alamos, 21 May 1951.

The following tabulations give results of all runs. The results of initial run are shown up to the point where plastic valve failed. All values given are referred to LST of 1320, May 20, 1951.

INITIAL RUN

PRECIPITATION

FEED:	DISSOLVER SOLUTION		12,840 curies
LOSSES:	METAL WASTE - 1st EXTRACTION	23 curies	
	METAL WASTE - 2nd EXTRACTION	28 "	
	METATHESIS	334 "	
	A-9 RINSE	298 "	
		683 curies	683 curies
YIELD:	94.6% BASED ON LOSSES		12,157 curies

[redacted]

RESIN CUBICLE

FEED:	METATHESIS FROM A-9 (analysed in feed tank)		12,157 curies
LOSSES:	FEED EFFLUENT	25 curies	
	FEED RINSE	29 "	
	VERSENE WASTE	53 "	
	HCl WASTE	88 "	
		195 curies	195 curies
*YIELD:	98.3% BASED ON LOSSES		11,962 curies

*At this point the mechanical failure of PV-23, a distribution valve, occured and the product was lost to the waste. No confirmation of this yield could be made but it is reasonable to assume that such a yield could have been expected.

RECOVERY RUN NO.1

PRECIPITATION

FEED:	(1) RETURN FROM NO.200 RESIN CUBICLE	11,962 *1	
	(2) INITIAL METATHESIS LOSSES	334	
	(3) A-9 RINSE - INITIAL	298	
	(4) DISSOLVER HEELS	291	
		12,885 curies	12,885 curies

LOSSES:	METAL WASTE EXTRACTION	1216	
	HEELS	844	
	A-9 RINSE	5950 *2	
		8010 curies	8010 curies

[redacted]

YIELD: 37.9 % BASED ON LOSSES		4875 curies

*1 Assumed that all product stripped from the column.
*2 A-9 rinse showed a ppt - incomplete metathesis.

RESIN CUBICLE

FEED: METATHESIS FROM RECOVERY RUN NO.1 (analysed in feed tank)		3869 curies
LOSSES: FEED EFFLUENT	151 curies	
FEED RINSE	382 "	
VERSENE WASTE	1 "	
HCl WASTE	77 "	
	611 curies	611 curies
YIELD: 84% BASED ON LOSSES		3258 curies
69.5% BASED ON YIELD (product sample)		2640 curies

RECOVERY RUN NO.2

PRECIPITATION

FEED: A-9 RINSE FROM RECOVERY RUN NO.1		5950 curies
LOSSES: METAL WASTE EXTRACTION	250 curies	
METATHESIS	116 "	
A-9 RINSE	1354 "	
	1720 curies	1720 curies
YIELD: 71% BASED ON LOSSES		4230 curies

[redacted]

RESIN CUBICLE

FEED: METATHESIS FROM RUN NO.2 (analysed in feed tank)		1305 curies
LOSSES: FEED EFFLUENT	1114 curies	
FEED RINSE	20 "	
VERSENE WASTE	41 "	
HCl WASTE	11 "	
	1186 curies	1186 curies
YIELD: 9.1% BASED ON LOSSES		119 curies
39.4% BASED ON YIELD *3		514 curies

*3 Product yields from recovery run No.1 and No.2 combined in product tank. The value given here is obtained by difference between yield for run No.1 and combination of run No.1 and No. 2.

FUMING NITRIC ACID PRECIPITATION

FEED: PRODUCT YIELD FROM RUN NO.1 and NO.2	3204 curies
LOSS: FUMING NITRIC WASTE	25 curies
YIELD: 98% BASED ON LOSSES	3179 curies
97% BASED ON YIELD	3157 curies

[redacted]

[redacted]

2.0 Valve failure - other operational difficulties.

The valve which failed was PV-23, a 5-port 4-way distribution valve. The valve is constructed of a stainless steel hull, a fluorothene body, and a teflon plug. The plug is spring loaded from the bottom which provides the pressure to seat the tapered plug in the body. The rotation of the valve plug is obtained thru a worm and gear mechanism mounted at the top of the valve. This mechanism is acuated by a flexible cable from the valve position indicator handle mounted on the panel board. Fig.1, gives a sectional view of the construction and assembly of this valve.

The exact nature of the failure has not been determined. The first indication that the valve was not satisfactory was derived from the fact that the liquid level in the main waste rose and with no apparent reason. A check of the position indicator showed that the flow should have been directed to the product evaporator. The first thought was that the position indicator was out of adjustment, however, a quick test revealed that the flow from the column was directed to the waste tank no matter what position was indicated. From this test it was concluded that the valve plug could not be made to rotate.

Failure of the valve plug to rotate could be attributed to a number of things; namely, the gear pin had sheared, the cable had parted, or the gears had stripped. The pin which fastens the driving gear to the gear shaft may have sheared, but this appears unlikely since all these pins are of hardened tool steel.

[redacted]

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That the cable had parted or broken loose from its end fittings is rather remote since all the cable assemblies were tested at 35 inch-lbs of torque prior to final assembly with the valve. Also an attempt to pull the cable from within the casing were of no avail. If the cable had parted it should be easily removed from the casing since it turns freely within the casing during operation. The fact that the casing has a number of sharp bends in it between the valve and the position indicator handle on the panel board would not interfere with the removal of the cable by itself or a portion of the cable; but, one would find it difficult to remove the cable with the end fitting attached because this fitting is too long and rigid to permit it to pass around too sharp a bend in the casing. If the gears had stripped a grinding or grating sensation would be felt in turning the position indicator handle; but instead, the indicator handle continued to turn as would normally be expected.

During the period when this valve - and all the plastic valves for that matter - were being designed and constructed, speed was of great importance and no long term rigid and thorough test program could be justified. Bearing in mind that the valve had been used numerous times prior to the time of failure, it is difficult to pin-point the exact cause of failure. When the cubicle has been decontaminated, a closer inspection of the valve and its related parts will be made to determine the following: How the valve failed, why it failed, and what should be done to reduce the chance for future failure. A memo to cover the above will be issued at that time.

[redacted]

Other operating difficulties arose and although they did not affect the process efficiency they are undesirable and should be eliminated.

A solution back-up occurred in the process filter cubicle and valve 33 between the process blow tank and process filters got hot. This valve is located outside the filter cubicle.

The sparger lines to the feed tanks in the resin cubicles got "hot" from a solution back-up.

At various places radiation was detected along the openings between the filter plugs and plug holes in both the crud and process filter cubicle.

Valve 208, a plastic diaphragm valve which regulated the flow from the eluate tank to the column, leaked air into the eluate stream. This leakage of air is undesirable since there is a possibility of "vapor-locking" the column.

The area adjacent to the funnel valves was high in background - approximately 5R/HR.

Radiation was detected along the space between the resin cubicle top and the cubicle pit.

During the evaporation to dryness of the product the loading cubicle and carrier became inexplicably contaminated.

This briefly covers the during-run operational difficulties. Two after-run difficulties arose and require special mention.

After the product was shipped attempts to wash out the heels loss in the product evaporator and recover this in a spare cone for isotope shipments were made. In this operation the cone manipulator failed. No carrier was available in which to place the cone in order to disengage it from the manipulator head. A pair of tongs was used to clamp the cone and the head rotated to disengage. The head however could not be made to rotate. Since the head is acuated by a flexible shaft from a dial knob on the control panel, it was assumed the cable had parted. Thin strips of lead were wrapped around the head and the movable shield rotated to cover the charging heads, this reduced the background to allow enough time for repairs. Close inspection revealed that the failure occurred where the drive shaft was brazed to a brass driving gear. The gear and shaft were removed and in brazing together again an attempt was made to fill the cable socket with brazing material in order to give the maximum joint strength. The manipulator now works very well. The alignment of the manipulator with the product cone carrier was not disturbed.

During decontamination another plug valve, PV-21, failed. This failure was that the shaft had parted approximately two to three inches from the position indicator handle on the panel board. A small portion of the cable left in the end fitting was sweated out and the cable resoldered to the end fitting. The cable casing was reduced in length to correspond to the new cable length. The valve has been used numerous times since repairing and works very satisfactorily. No explanation could be offered as to why the cable parted. The condition of the valve will be given along with the memo covering PV-23, the distribution valve which failed.

3.0 Plans are to decontaminate the No.200 resin cubicle, remove it from the pit, determine the exact nature of valve PV-23 failure, and make the necessary repairs to place the original equipment in an operable condition. Efforts will be made to reduce the radiation hazards along the cracks around the filter cubicles and the resin cubicle. All minor operational difficulties will be eliminated.

3.1 Several ideas have been advanced concerning the future plans. Changes in the process in which the hydrochloric acid concentration has been reduced from 6N to 1N would tend toward the use of stainless steel type 316, since this particular type of stainless has shown very good corrosion resistance properties at higher concentrations of HCl than is now used. With the cooperation of the laboratory group a series of lab scale runs will be made using a type 316 stainless column to determine the feasibility of using the material thru-out the cubicle.

If such proves feasible then the use of a metal plug in the present plug valves could be justified. This offers a definite advantage over the use of a plastic plug - as now practised, in that only one substance would be subject to flow under the sealing pressure. The body would tend to resolve itself around the plug, offering good sealing characteristics, and leaving the plug free to turn with a minimum of torque.

In the case the use of stainless is not feasible the plug for the plug valve could be made from Hastelloy "C" material which is known to have excellent corrosion properties to HCl concentrations above the present flowsheet conditions. The only objection to the use of Hastelloy "C" is the time delay in procurement.

[redacted]

It is possible to eliminate altogether the use of HCl in the process. The HCl is used to remove the excess sodium ion from the column which otherwise would be removed with the product. This contamination of the product is undesirable. However, a revision of the product evaporator or a purchase of a new one with revisions incorporated would make it possible to remove the sodium from the barium in the tank by fuming nitric acid precipitation step. The sodium ion concentration is such that a crystallization of sodium nitrate is experienced in the evaporation step proceeding the fuming nitric precipitation.

By raising the dip-legs they would not become plugged during this procedure and process control could still be maintained thru the liquid level indicator as is now practiced. Revision of the present product evaporator is impossible since this tank cannot be dismantled. Purchase of a new tank incorporating new dip-legs would require a long time (three to four months). On this basis this proceedure is not practical.

The plug valves could be eliminated and replaced with enough standard globe and gate valves to give desired process flow requirements. This proceedure would require major process changes and more flexible cables.

The use of the now existant HCl waste tank as a catch tank and subsequent jetting of each solution to its required tank is possible. This tank is made of Hastelloy "C" material and contamination of product with undesirables such as iron, chromiun, and others is not too serious. However, cross-contamination of eluate solutions is possible.

[redacted]

A spray wash preceeding the product removal step does not offer ultimate in safeguarding against this anticipated cross-contamination. Also this requires major equipment changes. However, such an equipment change does away with many remote valves and utlizes jets which seemingly are rather trouble-free. It would be well to mention here that attempts were made to titrate the column in No.200 cubicle to determine adequacy of the resin for this radiation level. No conclusive results were obtained and laboratory did not offer a definite statement regarding this point. This can only be answered by the successful completion of a run of the same radiation magnitude. In view of this stand, operations desires only to place the equipment in a satisfactory operable condition and this plan will be followed.

The engineering department is studying the use of metal plugs in the present valves; actually the repair of the present valves will be a replacement with new ones. The valves now in use are expected to be too hot to work on. A new radiation monitor will be ordered as well as a flow element for the induction flowmeter. In decontaminating it is highly probable that these instruments will be left inoperable.

Decontamination of the No.200 resin cubicle is well underway and is proceeding very satisfactorily.

Design and procurement of necessary material for repairs are in progress.

Los Alamos tentatively sets 15 July 1951, as next desired shipment date. This date can be met.

RHV:hh

[redacted]

re: RUN 43

ORNL
CENTRAL FILES NUMBER
51-4-36

This document consists of 4 pages.
No. 1 of 3 copies. Series A.

DATE: April 19, 1951

SUBJECT: Low Level Break-in-Run
706-D Modification Project

BY: Wm. E. Unger

TO: F. L. Culler

INV. 64

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RE[redacted]D DATA
Th[redacted] data as defined in the Atomic Energy Act [redacted] 1946.

[redacted]

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By Authority Of ____
By [illegible signature] Date AUG 31 1971

DATE: April 19, 1951

TO: F. L. Culler

FROM: Wm. E. Unger

SUBJECT: Low Level Break-in-Run - 706-D Modification Project

Approximately 60 Cl slugs were dissolved and processed, the dissolving was begun 1 April 1951, and the product left the plant 6 April 1951. Some cold slugs were pushed from the pile by error, and the dissolver solution contained 248 curies of Barium, considerably less than was expected.

The crud filtration was slow (about 3 hours) but the process filtration was fast (about 1 hour). The feed solution was transferred to the ion exchanger cubicles and the pH adjusted quite conveniently. Hot solution backed up in the feed tank sparger line, and in the blow tank to process filter line, as should have been anticipated. Operating changes will prevent future repetition. All instruments worked satisfactorily except the liquid level telemeter which behave erratically throughout the run (a loose connection in the panel equipment).

The fuming nitric precipitation yield was low because of the small quantity of barium present. The wastes were reworked using a 0.5 gram chemical barium carrier.

Precipitation:

Feed: Dissolver Solution		248 curies
Losses:		
Metal wastes	2 curies	
Metathesis wastes	1 curie	
A-9 rinse	3 curies	6 curies
Yield:		224 curies

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Ted Davis 1/30/95
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Single review of CCRP-declassified documents was authorized by DOE Office of ... August 22, 1994.

[redacted]

[redacted]

Ion Exchange Column

Feed: Cubicle Feed		224 curies
Losses:		
Feed waste	2 curies	
Versene waste	0.014 curie	2.1 curies
HCl waste	0.1 curie	
Yield:		204 curies

Fuming Nitric Precipitation

A. Feed: Nitric elution		204 curies
Loss: Fuming Nitric		-
Yield*:		58 curies
B. Feed: Loss (from above)		
Loss: Waste heel	18 curies	
Evap. heel	12 curies	30 curies
Yield:		106 curies
Total Feed:	204 curies	
Loss:	30 curies	
Yield:	164 curies	

Overall

Feed:	248 curies
Losses:	38 curies
Yield:	164 curies

*Product Analysis

Sr	0.05 curies
Pb	4.2 mg
Ce	$9.3 \cdot 10^{-4}$ curies

[redacted]

[redacted]

Yields

	Based on Loss	Based on Yield
Precipitation	97.5%	90.7%
Ion Exchange	99.0%	91.0%
Fuming Nitric	83.7%	80.5%
Overall	81.3%	66.0%

The product drying was slow, even slower than expected. Apparently, the conduction of heat from the point of hot air entry at the top of the product cone, down the cone walls, and into the liquid at the bottom, has a considerable influence upon the drying rate. The platinum liner of the cone and the annular air space it forms reduces the heat transfer into the liquid. Los Alamos will be requested to sweat the liner to the cone with silver solder brazing alloy.

The loading mechanism worked well, and the 164 curies of product was shipped at 1700 6 April 1951; arrived at Los Alamos at 2300 8 April 1951.

Wm. E. Unger

Wm. E. Unger

[redacted]

VII 3a (3)

51-6-181
FLS-740

This document consists of 7 pages.
No. 3 of 7 copies,
Series FLS.

re: RUNS 43/44

TO: F.L. Steahly

FROM: W.E. Unger

DATE: June 15, 1951.

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~~TO~~
By Authority of [signature] AUG 31 1971
Name Title Date

PART NUMBER V

QUARTERLY REPORT

Ted Davis 1/30/95

TITLE: 706-D MODIFICATION PROJECT

WORK BY: T.A. Arehart, G.B. Berry, B.F. Bottenfield, R.V. Foltz, E.M. Shank, W.E. Unger and R.K. Vaughan.

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David R Hamrin 1/30/95
Technical Information Officer Date
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[redacted]

The 706-D Modification project covered the development of a process, and the development, design, and installation of equipment to improve the yield and quality of the 706-D Rala plant. The project included the addition of a dissolver solution clarifying filter and a process filter to replace the former separation by decantation, the installation of duplicate ion-exchange purification equipment housed in floor pits, and the installation of product charging facilities to accommodate the product carrier adopted by Los Alamos.

COSTS

The charge account (3370-36) was closed as of 26 May 1951. The costs as of that date had totaled $ 270,000.00, against an estimate (Unger to Stringfield, 19 January 1950) of $ 287,400.00, which was budgeted (Scharsten to Stringfield 28 March 1950) as project funds. The cost increases occasioned by additional design complexities, equipment delivery delays, and competition with other urgent and high priority projects were more than off-set by savings realized from the use of reclaimed shielding lead made available by the Operations Division personnel.

BREAK IN RUNS

The equipment was tested through a series of cold runs, one 200 curie run, and one full-scale 15000 curie run.

200 CURRIE RUN

Approximately 60 Clinton pile slugs were charged 6 April 1951. The dissolver solution clarification filtration was slow (about 3 hours, a defective weld in the filter suction line was discovered and has since been repaired) but the process filtration was fast.

A back-up from the filter to the blow-down line valve indicated the need for shielding around the valve. Hot feed solution or vapors backed up the cubicle feed tank sparger line, indicating the need for a constant purge flow of air thru the sparger line.

The ion-exchange run proceeded satisfactorily until the final nitric acid precipitation step. Apparently, the gram barium content was insufficient to exceed the solubility, and the product passed thru the filter to the waste. The waste was returned and reprecipitated satisfactorily with 500 milligrams of cold barium as a carrier.

[redacted]

[redacted]

The final evaporation in the shipping cone was slow, because of the poor heat transfer of the lined shipping cone. This has been confirmed by Los Alamos and the liners for replacement cones are being assembled by brazing.

FULL-LEVEL RUN #44

Approximately 77 1800-gram Hanford slugs were charged 13 May 1951. The dissolver solution clarification filtration and product filtration were fast (2.6 gpm and 1.5 gpm respectively). Hot solution back-up into the filter blow line and the ion-exchange feed tank sparger proved troublesome despite the precautions exercised by the operators. The blow line valve will be surrounded with additional shielding and the feed tank sparger line control valve will be moved close to the cubicle shielding.

The run rpogressed essentially without incident and with splendidly low losses until the product was eluted from the ion-exchange column. A plastic plug valve failed (a failure of the mechanical drive someplace between the valve plug and the panel board) in position to conduct the column effluent to the waste tank only. The product, with the accummulated wastes, was re turned to "A" cell and reprocessed (with extremely high losses) through the spare ion-exchange equipment with a yield of about 20%.

The yields in curies from the two hot runs were as follows:

	LOW LEVEL		FULL LEVEL	
Dissolver Feed	248		14509	
Sulphate Precipitation	224	90.3%	13888	96.5%
Ion Exchange Column	204	82.3%	3620*	24.9%*
Fuming Nitric	164*	66.2%*	3593*	24.7%*

* Reprocessed

INSTRUMENTATION

The Liquid Level Telemeter has worked well, about as predicted by the inventors. It is considered a useful check against the manometer but it characteristically is neither as dependable nor as accurate as the manometer. The telemeter accuracy is influenced by the relative conductivity of the solution (it behaves erratically on clean tap water, for instance), its reliability is reduced to that of its relatively vulnerable electronic circuits, and its life is limited to the radiation damage tolerable by the germanium diode element which for technical reasons must be located in proximity to the tank unit.

[redacted]

[redacted]

The induction flowmeter has proved to be a very sensitive indication of flow rate. It is apparently influenced but very little by the conductivity of the stream (the conductivity of the stream must be large compared only to the insulating medium of the canula element, in this case, glass), and not at all by the density or viscosity of the stream. It was suspected that the deposition of foreign matter in the canula tended to form an orifice, with correspondingly higher flow velocities and indicated flow rate than was actually the case. This should be minimized, even at the sacrifice of sensitivity, by using as large a diameter canula as possible. The process unit appeared to be rugged and reliable, and its life is limited only by the radiation damage sustained by its gaskets and the electrical insulation on the magnet windings and electrical leads to the panel board.

The Moore differential transmitters have worked well; however, the bellows elements do take a slight permanent set with each substantial pressure change, with a corresponding annoying shift in the zero point. The panel gauges exhibited the tendency of gauges in general to display "friction-lag", making their readings appreciably less sensitive than the manometers with which they were compared.

SHIELDING

The bulk shielding is ample. Some "shine" from the space between the cubicle plug and the adjacent channel wall will have to be plugged with lead sheet. All other radiation was contributed by the back-up of process solutions into unshielded lines.

EQUIPMENT DESIGN

The operating difficulties that occurred during test runs and that are attributable to design features of the equipment are listed below:

Small lines

Minimum-sized process lines were selected because of the relatively low flowrates required and the expense of the tantalum tubing required by the product purity specifications. The lines have been plugged by welding scale, flakes sluffed from the teflon plug valves, and other unidentifiable foreign matter, even though the tanks and equipment were assembled with care and copiously washed.

The manometer lines were also of minimum-size, which increases the air purge pressure drop. Larger lines would have minimized the aggravation of the purge rate influencing the manometer reading.

[redacted]

[redacted]

Pressure feed

It is always advisable to avoid using a process vessel as a vacuum or pressure pot, and some operational difficulties were expected were this to prove necessary.

The feed tank was located at the top of the cubicle and the receiver at the bottom to obtain as great a gravity head as possible. Pressurized feed was necessary and the change from a low pressure to a higher pressure induced the back-up of solution into all unpurged lines that dipped below the liquid level (the feed tank sparger was the principal offender).

All gravity feed would have required cubicle space considerably in excess of what was available.

COLUMN SYSTEM

The column must be capable of being charged with resin added thru a funnel from outside the shielding; of being discharged by back flushing to a hot waste system; of accommodating process flow from inside the cubicle and eluate (cold) from without the cubicle. A valving arrangement was required but product purity specifications precluded the use of standard commercial valves, and special valves were designed and fabricated of the chemically inert poly-fluoro plastics. The physical characteristics of the plastics limited the valve port diameters to sizes too small to pass unslurried resin readily. As a result, operators must observe certain techniques in charging the resin slurries, adding to the complexity of operation.

The nature of the ion-exchange resin requires that the resin be always submerged. Any gases introduced into the system (by the radiation-induced dissassociation of water, by the release of air dissolved in the elutrients, or blown into the column at the end of a pressurized feed) accummulates in the column and gradually displaces the liquid level in the column which then eventually recedes below the level of the ion exchange resin. There is no operational indication of when this has occurred, and operators are forced to interrupt the process periodically, change the valve settings and allow any gases that may have accummulated to be displaced upward into the deaerator. Although with careful operation this is probably not necessary. The column is "bled" at least once in each run. This has increased the complexity of operation.

The column system should be provided with a means for determing the resin level at the beginning of a run, and the column liquid level continously.

[redacted]

[redacted]

The specially-designed plastic plug valves are highly desirable from a process standpoint in that they minimize cross-contamination and the time required for valve settings. The plugs are driven by a worm and pinion, which in turn is driven by a flexible shaft extending from the valve to the panel board control handle and valve position indicator. The length of the flexible shaft and the torque it is required to transmit, combine to produce considerable (ca. 15°) torsional deflection, referred to as "whip" or "back lash". The effect of the whip on the valve positioning is small because it is reduced by the worm and pinion drive by a ratio of 40:1, but the "springiness" of feel is disturbing to the operators who would have preferred a solid shaft drive.

The plastic valves, being dimensionally unstable, tend to flow over long periods of time to relieve concentrated stresses. The resulting deformation adversely affects the seal of the plug and the ease with which it is operated.

TANK DESIGN

The process tanks should have been equipped with inter-transfer jets for emergency use, with spray heads for rapid tank wash-downs, and with specific gravity manometer probes. These lines were held to a minimum because of space limitations.

The tantalum evaporator manometer will read down to 500 ml volume. This is frustrating to the operators who must dissolve the final product ina maximum of 300 ml of water.

SAMPLERS

The samplers operate fairly satisfactorily. The principal complaint is the special technique and "feel" that is necessary in positioning the sample receptacle under the sample valve. The samplers are mechanical contrivances and depend for their sucessful operation upon the nicety of construction. The slight differences in fabrication impart to the samplers unlike characteristics that is annoying to the operators who strongly prefer rugged simple equipment that functions very positively.

FINAL EVAPORATION

The final evaporation that takes the product to dryness in the platinum-lined shipping cone was expected to take place in about six hours. All equipment development and installation tests were made with a brass dummy in lieu of the actual shipping cone which was not then available. The lower heat transfer across the platinum lining so decreased the evaporation rate that twelve hours were required to take the product to dryness. Future cones will be joined to the liners by silver brazing.

[redacted]

The charging heads are designed to avoid turbulence of the hot air stream to minimize the loss of product by "dusting". The flow of hot air is induced by the vacuum on the cone so that any leakage between the cone and the charging head would be a leakage into the off-gas system, not out into the loading cubicle. Despite this design precaution, the cubicle and carrier became perceptibly contaminated; however both were easily decontaminated with a water wash.

CONCLUSION

The mechanical failure of the process valve resulted in a non-standard run from which no positive conclusions can be drawn. All indications are that the ion-exchange process chemically accomplishes its purpose. The failure of the last run is attributable solely to equipment failure.

126

Return to CF

[redacted]

ORNL
CENTRAL FILES NUMBER

51-8-5

re: Run 45 W slug activity

This document consists of 2 pages.
No. 1 of 5 copies. Series A.

DATE: August 1, 1951

SUBJECT: Discussion of Next 706-D RaLa Run

BY: Wm. E. Unger

TO: F.L. Culler

INV. 64

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Page 2.

F.L.Culler

W.E.Unger

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By DM Date AUG 31 1971

Los Alamos has asked E.J.Witkowski to ship 5000 curies on the next run (scheduled to start 12 August 51), and LA has indicated that 30,000 curies will be requested sometime late in September.

EJW wants to produce 10,000 old ORNL curies (14,500 absolute curies) on the 12 August run and has obtained LA's approval.

W slugs now contain considerably more barium than a year ago due to a flux increase of 50% to 80%. Using past D-building dissolver solution analyses, the minimum barium content will be 132 to 165 available absolute curies (assuming six day process time plus four days shipping time). Each carrier holds 39 slugs, so that at least three carrier loads would be required, and preferably four, to insure enough feed. This would require three to four dissolvings and sulphate precipitations.

There is a distinct advantage in making as few sulphate precipitations as possible per ion exchange run. Some lead carrier is lost by solubility on each precipitation, (assumed to be 20%), and this amount is added each time. But the actual solubility loss is not known and even appears to vary from run to run. Obviously, the greater the number of precipitations the greater the cummulative departure from assumed lead concentration. The ion exchange process can only tolerate a 30% deviation. Lead analysis of the column feed is uncertain and time consuming but will be necessary for all runs of more than two sulphate precipitations; so that the selection of slugs of maximum barium content will be of advantage.

Hanford is interested in keeping the metal diverted from production channels to a minimum. EJW has arranged with Hanford to specify only the barium content required at time of arrival at Oak Ridge. Hanford will then select and ship slugs sufficient to provide the feed required by EJW.

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Single rereview of CCRP-declassified documents was authorized by DOE Office of Declassification memo of August 22, 1994.

Wm. E. Unger

Wm. E. Unger

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RUN 45

ORNL B-13
CENTRAL FILES NUMBER
51-8-146

Date August 20, 1951

Subject Analytical Data - Run #45

By E. I. Wyatt

To

File

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[redacted]

To: M.E. Ramsey
From: E.I. Wyatt

Analytical Data
Run #45
LST 1400 8-16-51
Shipment #52

8-2[illegible]51

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By [illegible] Date SEP 01 1971

Batch	Curies	Slugs	A-8	% Loss
A	9257	41.2	19	0.07
B	8151	41.9	27	0.09
C	7087	39.9	97	0.34
D	3913	24.2	—	—
	28408	147.2	143	0.50

Other Cell A Losses	Curies	% Loss
Metathesis Losses (8WCW)	427	1.50
A-9 Rinse	427	1.50
* ½ IMC	3544	12.47
* IMD	3913	13.77
Total Cell A Losses	8454	29.75

Extraction Cell Losses	Curies	% Loss
Feed Effluent (MWFE)	659	2.32
Sr. Extraction (ITNAVE)	218	0.77
HCl Wash (MWHClE)	396	1.39
Feed Tank Rinse	6889	24.25
Fuming Nitric Waste	1570	5.53
PE Rinse	157	0.55
Column Holdup #1	650	2.29
Column Holdup #2	20	0.07
Total Extraction Cell Losses	10559	37.17

	Curies	% Loss
Total Losses Accounted for	19013	66.92
Product		
PE-1	10449	36.78
Total Product shipped (Radiochemical)	8722	30.70
Material Balance	27735	97.63

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* ½ of Batch C and all of Batch D were not added to the Run [redacted]

cc:
M.E. Ramsey
E.I. Wyatt
E.J. Witkowski
Central Files

[redacted]

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X-426 (Revised 1-52)

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ORNL
CENTRAL FILES NUMBER
52-5-168

RUN 46
– new ion exch. process
– transport by air

COPY NO.

"This document consists of 5 pages
No. 9 of 9 copies, Series A

DATE: May 20, 1952

SUBJECT: Quarterly Report - 706-D RaLa CT-16
Report Period: 2/10/52 to 5/10/52

TO: F.L. Steahly

FROM: Wm. E. Unger

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Chemical Technology Division
Process Design Section

Report Period: 2/10/52 to
5/10/52

To: F.L. Steahly

From: Wm. E. Unger

QUARTERLY REPORT

CT-16

Subject: 706-D RaLa

0.0 Abstract

A 23000-curie run was shipped to Los Alamos in January. An insoluble crud, removable by caustic precipitation, interfered with final processing at Los Alamos. No equipment changes will be made at ORNL until confirming runs have been made.

1.0 Summary

The product of run 46 (23000 curies) was processed with difficulty by Los Alamos because of the presence of an insoluble crud that was finally removed by caustic precipitation. Removal of the crud, if it occurs again, is necessary before the lanthanum can be utilized. The crud could be removed at Oak Ridge before the barium is shipped, or at Los Alamos, in which case the accumulated lanthanum would be sacrificed with the crud. The accumulated Lanthanum is of value, because of cerium growth, only if the total transportation and handling time between Oak Ridge and Los Alamos does not exceed 40 hours.

Air transportation of the barium product from Oak Ridge to Los Alamos is being fostered by the Los Alamos - AEC who have procured a suitable airplane and adapted it to support the concentrated weight of the Los Alamos Carrier. Whether or not air transportation is justifiable depends upon a balance of the economic value of the extra milking, off-set by the additional cost of air transport and the cost of caustic precipitation equipment at ORNL. No equipment changes will be made until:

a. the presence of the crud is confirmed by a second run,

b. air shipment becomes a reality, and

c. the future RaLa requirements are firmly forecast.

One of the two Tucson (143 slug) Carriers has been shipped to Hanford for testing at that site.

2.0 Introduction

The 706-D Modification project covered the development of a process and the development, design and installation of equipment to improve the yield and product quality of the 706-D RaLa plant. The process was changed to replace the old HCL-ether and fuming nitric acid precipitation processing with an ion-exchange absorption process for purifying the RaLa product of its chemically similar contaminants. In addition to the installation of duplicate ion-exchange purification equipment housed in floor pits, a dissolver solution clarification filter and a process filter were installed to replace the former separation by decantation. New facilities, also housed in floor pits, were installed to accommodate a new product carrier adopted by Los Alamos and in which the product is shipped from Oak Ridge to Los Alamos.

On the 12th of January 1952 approximately 30,000 curies, contained in some 176 Hanford slugs, were processed through the D-Building RaLa equipment (Run No.46). The run proceeded successfully without incident through the final precipitation. For some reason not yet clearly understood the barium nitrate precipitate did not completely dissolve in the water added to transfer it to the shipping container, and some 17,000 curies was left behind. The insoluble residue, after a second nitric acid precipitation, dissolved readily in water and was transferred to the product container to join the first transfer of 6000 curies to make a total of some 23,000 curies shipped to Los Alamos.

3.0 Insoluble Crud

The separation of the desirable lanthanum daughter of radioactive barium is called "milking." The first milking at Los Alamos of the product of Run No. 46 was rejected for the cerium content, but the second milking contained a gray, almost gelatinous, precipitate that displayed about the same density as that of the liquid. The gray precipitate was not soluble in fuming nitric acid, and although it centrifuged well, it readily floated back into suspension on the slightest disturbance of the supernate, which is separated from the precipitate at Los Alamos by decanting.

After repeated and unsuccessful attempts to separate the crud from the product solution the barium product residue was treated with caustic employing lanthanum for a carrier. The precipitated lanthanum apparently carried down the gray crud and the barium solution was then successfully decanted from the accumulated lanthanum and gray crud.

The identity of the gray crud is of course not known. It may be decomposition products of Versene or traces of organic from the decomposition of the resin used in the ion-exchange separation of barium from its contaminants. The crud, regardless of its identity, is objectionable to Los Alamos, and must be removed before the lanthanum can be utilized at Los Alamos.

It may be necessary to install equipment at Oak Ridge to carry out a caustic precipitation to separate the gray crud from the product before it is shipped to Los Alamos. Presumably the gray crud could be removed at Los Alamos as well as at Oak Ridge, but at the expense of the lanthanum that will have grown in by the decay of barium during the transportation of the product from Oak Ridge to Los Alamos. At the present time this lanthanum must be discarded anyway, because of the cerium growth that exceeds tolerance in the final product, but in the event that air transportation of the product from Oak Ridge to Los Alamos becomes feasible, it would probably be economically feasible to install the equipment at Oak Ridge to remove the crud from the product before shipment, so that the lanthanum growing in during transportation time could be utilized on arrival at Los Alamos.

It was decided that no equipment modification would be made at ORNL to remove the objectionable crud until:

1. the presence of crud in the product solution is confirmed by a second run,

2. until air shipment becomes a reality, and

3. until some commitment on the future of the RaLa program is available either from the consumer or the AEC.

3.1 Air Transportation

The shipment of the product from Oak Ridge to Los Alamos by truck-drawn trailer requires about two or three days and is relatively expensive because of the overtime pay drawn by the guards and the truck drivers. The AEC, responsible for the actual transportation of the product, suggested air transportation about 18 months ago, and the Los Alamos Carrier was designed with air transportation in mind. The Los Alamos AEC procured an airplane and altered its fuselage to distribute the weight of the 4-ton Los Alamos carrier, and one or two air shipments of the empty carrier were made. The airplane was then lost in an air collision at the Sandia Airbase about a year ago and air transportation of the RaLa shipment was temporarily dropped. The AEC has now procured another airplane and has again made the necessary structural changes in its fuselage to adapt it to the transportation of the Los Alamos carrier.

Whether or not shipments are made by air is quite immaterial to Los Alamos and to us, but will be supported by all concerned for the economic gain to be realized if shipment can be made from ORNL to Los Alamos within the 40 hour allowable growth period for the cerium contaminant specification, and if caustic precipitation successfully eliminates the crud at ORNL. This gain amounts to being able to utilize three milkings per shipment instead of two. Assuming that the 706-D Building direct run costs range between $6000.00 and $10,000.00, the value of the third milking would amount to about $3000.00, and this gross gain should be reduced by the additional cost of air shipment over that of truck shipment plus the amortizied cost of the modifications in 706-D Building equipment required to eliminate the objectionable crud before shipment from ORNL. It is apparent that the net gain will not be large.

3.2 Tucson Carrier

The Tucson Carrier, procured by the Operations Division to increase the capacity of slugs shipped from Hanford to ORNL, were found to have been partially welded with black iron welding rods. The objectionable welds have been repaired and the finished Tucson Carrier shipped to Hanford for testing and development work at that site. The irradiated slugs, feed for the next hot run presently scheduled loosely for the next month or so, will be shipped in the Tucson Carrier.

4.0 RaLa Future

The 706-D Building RaLa equipment has always been based upon an expected run frequency of approximately 10 runs per year, or about one per month, but at no time in the history of D-Building have more than seven runs been made in any one year. A great deal of emphasis has also been placed upon the relative reliability with which a run should be produced at any specified time. But from past experience the Operations Division is growing skeptical of the urgency of the schedule issued by Los Alamos; for one thing runs that have been shipped on an ostensibly tight schedule have waited at Los Alamos for a week or more before receiving their first milking, and on some occasions delays of a week or so in a given run schedule have not been at all perturbing to the consumer at Los Alamos. The MTR-B RaLa project, which was initiated on the basis of a need for a greater frequency of high-level RaLa sources low in cerium and inert barium content, was abandoned because no information on future schedules or requirements of RaLa could be obtained sufficiently firm to justify the MTR-B project expense and manpower consumption.

It has now been pointed out that RaLa shipments of the size presently foreseen can easily be accommodated by the 706-D Building equipment at ORNL. For the time being, slugs irradiated at Hanford can be used, and later, slugs irradiated at Aiken. The amount of money, time and effort in research and development that should be expended to perfect the process and equipment at ORNL depends to a great extent upon the relative longevity of the RaLa requirements. The future of RaLa requirements originally was to have been clearly stated by the AEC in March of this year, but this decision has now been postponed until September of this year, probably to await certain Fall tests presently planned by the AEC.

At the present time the demonstrated instability of RaLa schedules and the present uncertainty of future requirements for RaLa make it virtually mandatory that no process equipment changes be undertaken until relatively firm schedules and future requirements are available.

B. F. Bottenfield for
Wm. E. Unger

WEU:hh

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C-2
RUN 46

ORNL
CENTRAL FILES NUMBER
52-1-131

Date January 22, 1952

Subject Analytical Data - Run #46

By E. I. Wyatt

To M. E. Ramsey

File

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To: M.E. Ramsey

From: E.I. Wyatt

Analytical Data
Run #46
LST 0500 1-19-52
Shipment #53

1-21-52

Batch	Curies	Slugs	A-8	% Loss
A	7890	39.4	12	0.04
B	7002	38.9	14	0.05
C	6989	39.3	20	0.06
D	5553	36.7	7	0.02
E	3407	22.1	22	0.07
	30841	176.4	75	0.24

Other Cell A Losses	Curies	% Loss
Metathesis Losses (SWCW)	148	0.48
A-9 Rinse and Prod. Filter Rinse	1132	3.67
	1355	4.39

Extraction Cell Losses		
Acetate Feed Effluent (ITFE)	0	0.00
MWNaOH-E	17	0.05
Feed Effluent (MWVFE)(Versene)	2	0.01
Sr Extraction (ITNaVE)	13	0.04
HCl Wash (MWHCl-E)	3	0.01
Feed Tank Rinse	144	0.47
MWFN before Recovery	115	0.37
PE Rinse before Recovery	502	1.63
MWFN Recovery	98	0.32
PE Rinse Recovery	318	1.03
M.W. Resin Product Holdup	91	0.30
Total Extraction Cell Losses	1303	4.23
Total Losses Accounted for	2658	8.62

Product

PE-1	25728	83.42
PE-HNO_3 Rinse	18240	59.14
Product Calculated to be in carrier at 1-17-52	6871	22.28
(PE-1 less MWFN less PE H_2O Rinse less PE HNO_3 Rinse)		

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	Curies	% Loss
PE-2	16502	53.51
Product from 2nd Recovery (PE-2 less PE Rinse Recovery)	16184	52.47
Calculated Total Product Shipped 1-19-52 (2nd Recovery Prod. plus Residual in Carrier)	23055	74.75
Material Balance	25713	83.37

cc:

M.E. Ramsey
K.I. Wyatt
E.J. Witkowski
F.L. Steahly
R.E. Blanco
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RUN 47

OAK RIDGE NATIONAL LABORATORY
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ORNL
CENTRAL FILES NUMBER
52 6-34

COPY NO. 3a
E. J. Witkowski

DATE: June 9, 1952

SUBJECT: Analytical Data Run #47

TO: M. E. Ramsey

FROM: E. I. Wyatt

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To: M.E. Ramsey

From: E.I. Wyatt

Analytical Data
Run #47
LST 1940 -6-4-52
Shipment #54

6-9-52

Batch	Curies	Slugs	A-8	% Loss
A	6558	41.8	10	0.03
B	6033	39.7	8	0.02
C	6175	41.2	17	0.04
D	5190	33.5	309	0.81
E	6059	39.1	23	0.06
F	5399	37.2	44	0.12
1M Heels	2485	19.3	—	—
	37899	251.8	411	1.08

Other Cell A Losses	Curies	% Loss
SWW	3719	9.81
1M Heels	2485	6.56
SWCW	394	1.04
A-9 P.F. Rinse	939	2.48
Total Cell A Losses	7948	20.97

Extraction Cell Losses		
Acetate Feed Effluent (MWFE)	0	0.00
MWT NaOH	394	1.04
Feed Effluent (MWVFE)(Versene)	2	0.00
Sr. Extraction (MWNaVE)	3	0.01
HCl Wash (MWHClE)	4	0.01
Feed Tank Rinse	39	0.10
MWFN	822	2.17
P.E. Rinse	1023	2.70
Total Extraction Cell Losses	2287	6.03
Total Losses Accounted for	10235	27.00

Product		%
PE-1	29522	77.90
PE-2	28855	76.14
Total Product Shipped (Radiochemical)	27832	73.44
Material Balance	38067	100.44

cc: M.E. Ramsey
E.I. Wyatt
E.J. Witkowski
F.L. Steahly
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ORNL CENTRAL FILES NUMBER
52-7-29

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From: E. I. Wyatt

Analytical Data
Run #48
LST 1415 7-4-52
Shipment #55

7-7-52

Batch	Curies	Slugs	A-8	% Loss
A	7771	42.4	10	.03
B	7518	39.5	6	.02
C	6357	41.7	11	.03
D	6250	37.5	35	.10
E	5178	32.0	18	.05
F	2804	19.5	48	.12
	35878	212.6	125	.35

Other Cell A Losses	Curies	% Loss
5WC	694	1.93
SWCW	523	1.46
A9 and Pres. Filter Rinse	1421	3.96
Total Cell A Losses	2763	7.70

Extraction Cell Losses	Curies	% Loss
Acetate Feed Effluent (ITFE)	1	.00
MWNaOH	73	.20
Versene Feed Effluent (MWVFE)	64	.18
Sr Extraction (ITNaVE)	50	.14
HCl Wash (MWHClE)	56	.16
Feed Tank Rinse	17	.05
5WFE	2745	7.65
PE Rinse	1713	4.77
Total Extraction Cell Losses	4719	13.15
Total Losses accounted for	7482	20.85

Product		
PE-2	25035	69.78%
Product Shipped (Radiochemical)	23322	65.00%
Material Balance		85.85%

cc: E. I. Wyatt
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Run #49
LST 0825 8-3-52 [redacted] 8-4-52
Shipment #56

To: M.E. Ramsey
From: E.I. Wyatt

Batch	Curies	Slugs	A-8	% Loss
A	8060	40.4	54	0.13
B	7577	37.4	10	0.03
C	7362	39.2	7	0.02
D	6524	38.7	11	0.03
E	6338	38.5	26	0.06
F	4307	25.7	111	0.27
1M Heels	228	2.3	--	----
	40396	222.2	219	0.54

Other Cell A Losses	Curies	% Loss
1-M Heels	228	0.57
5 WC	515	1.27
8WCW	163	0.40
A9 and Proc. Filter Rinse	459	1.14
Total Cell A Losses	1584	3.92

Extraction Cell Losses

Acetate Feed Eff. (ITFE)	3	0.01
MWNaOH	50	0.12
Versene Feed Eff.(MWVFE)	21	0.05
Sr. Ext. (ITMaVE)	21	0.05
HCl Wash (MWHClE)	18	0.04
Feed Tank Rinse	5	0.01
8WFE	1278	3.17
PE Rinse	4010	9.93
Resin Column Hold-up	2264	5.61
Total Ext. Cell Losses	7670	18.99%

Total Losses accounted for	9254	22.91%

Product

PE-2	30625	75.8%
Product shipped (Radiochemical)	26615	65.9%
Material Balance	---	88.8%

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cc: E.I. Wyatt
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To: M. E. Ramsey
From: E. I. Wyatt

Analytical Data [redacted] 8-28-52
Run #50
LST 0615 8-28-52
Not Shipped

Batch	Curies	Slugs	A-8	% Loss
A	8818	20.9	44	
B	7906	20.5	15	
C	6697	20.1	70	
D	6277	18.3	27	
E	4774	14.7	33	
F	3513	11.2	168	
	37985	105.7	357	0.94
Other Cell A Losses				
SWC	292			
SWM(Recovery)*	83			
SWCW(Rec)	21			
A-9 and PF Rinse	197			
A-9 and PF Rinse(Rec)	378			
Total Cell A Losses	1328			3.50%
Extraction Cell Losses				
PE-2*	19185			50.51%
FT Rinse	3			
FT Rinse (Recovery)	54			
ITFE	9			
ITFE (Recovery)	1			
MWNaOH	150			
MWNaOH(Recovery)	6			
MWVFE	57			
MWVFE(Recovery)	1			
ITNaVE	25			
ITNaVE (Recovery)	1			
MWHClE	18			
MWHClE (Recovery)	1			
SWFN (Recovery)	4037			
PE Rinse #2	32			
PE Rinse (Recovery)	1352			
Resin Col. Hold-up	20			
Ext. Cell Losses	24952			65.69%
Total Losses Accounted for	26280			69.19%
PRoduct				
PE-2 (Recovery)	3557			
Product (Radiochemical)	2205			5.80%
Material Balance				74.99%

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* Material lost during and/or after final evaporation

cc: E. I. Wyatt
E. J. Witkowski
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CENTRAL FILES NUMBER
52-9-3

DATE: September 3, 1952

SUBJECT: Report of RaLa Run #50

RE: RUN 50

COPY NO. 2A

E. J. Witkowski

TO: Dr R. P. Hammond

FROM: E. J. Witkowski

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September 3, 1952

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Dr. R. P. Hammond
Los Alamos Scientific Laboratory
P. O. Box 1663
Los Alamos, New Mexico

Dear Phil:

John Schulte asked me to write a brief description of the difficulties we experienced in attempting to make the last batch for you. The following report is the one we submitted as a portion of the Oak Ridge National Laboratory Weekly Progress Report. I hope it will serve your needs.

(18) Thw

"Run #50, started on August 8, 1952, was a complete failure; no product was shipped. The main difficulties which accounted for the failure of this run were the plugging of the process filters, leaks in the process filter lines, leaking thermocouple well in the product evaporator and the loss of product from the shipping cone into the off-gas line during the product drying operation.

The filtration through the process filters became progressively slower with each of the six batches extracted. The rate became so poor in the first metathesis filtration that it became necessary to revert to the old method of decantation to remove the waste supernatant from the product precipitate. This step resulted in a product loss of almost twenty-five percent.

Another high loss of approximately twenty-five percent was experienced between the dissolving operation and the sampling of the product in the resin column cubicle feed tank. It now appears that this loss was caused by leaks around the process filter equipment. Similar losses, although never as large, had also been experienced in previous runs; they could never be accounted for by the analyses of the wastes.

The operations through the resin column cubicle appeared to progress with practically no difficulty. A reddish-brown color was observed in the product sample prior to the transfer to the product shipping cone, but at this point the run was too far behind schedule and there was too little product left to attempt to purify it.

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The batch prior to the transfer to the cone contained approximately 20,000 curies of product. After drying however, the radiation readings indicated that only about 8,000 curies were present in the cone. The shipping cone was placed back on the charging head while a search was made for the missing product. The search through the equipment proved futile and about eight hours later when the product radiation was again measured, it was discovered that practically all of the 8,000 curies which had been in the cone had also disappeared.

It was now evident that the dry product had been lost from the cone into the off-gas line. At this point, Los Alamos agreed to accept as low as 7,000 curies if it could be recovered from the 10,000 curies held by the metathesis and fuming nitric wastes.

The recovery of the wastes proceeded satisfactorily to the fuming nitric precipitation step prior to the transfer of the product to the cone. The product was again reddish brown. Approximately fifty percent was lost in fuming nitric waste, twenty-five percent was left behind in product evaporator and twenty-five percent was transferred to the shipping cone.

The small amount of product (2,000 curies) was too small to be of any use to Los Alamos so it was used to prove that it was possible for the product to be drawn into the off-gas line from the shipping cone. Approximately ninety percent of the dry product was again lost to the off-gas line by keeping it under the charging head for several hours and by repeating the movements the cone was put through during the main part of the run.

Further investigation after the run was discontinued revealed a bad leak in the thermocouple well which explained the presence of iron in the product. It is suspected that the presence of iron may have changed the physical properties of dry product in such a way as to enable it to be swept out of the cone into the off-gas line.

The next run is scheduled to be made early in October. It will be impossible to get the equipment repaired by that time but it is believed that a successful run may be made by making alterations to the operating procedures which will avoid the difficulties experienced in Run #50".

Very truly yours,

EJW

E. J. Witkowski

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X-426 (Revised 1-52)

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ORNL
CENTRAL FILES NUMBER
52-10-11

RUN 51

DATE: October 2, 1952

COPY NO. 2a

SUBJECT: Analytical Data - Run #51

TO: M. E. Ramsey

FROM: E. I. Wyatt

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A.E. Ramsey
A: E.I. Wyatt

Analytical Data
Run #51
LST 1550 9-30-52
Shipment #57

10-2-52

Batch	Curies	Slugs	A-8	% Loss
A	8070	18.48	36	0.09
B	7988	18.94	484	1.20
C	7721	20.42	159	0.39
D	6519	18.60	107	0.27
E	5596	17.54	133	0.33
F	4403	12.36	170	0.42
	40297	106.34	1089	2.70

Other Cell A Losses

	Curies	% Loss
SWC	4065	10.08
SWCW	1309	3.25
A-9 and PF Rinse	662	1.64
Total Cell A Losses	7122	17.67

Extraction Cell Losses

	Curies	% Loss
AS-Cubicle Wastes	505	1.25
Feed Tank Rinse	14	0.04
HCl Waste (MNHClE)	19	0.05
SWFA	445	1.10
PE Rinse	392	0.97
Resin Column Hold-up	11	0.03
Total Ext. Cell Losses	1386	3.44

CLASSIFICATION CANCELLED
ADD signature 1/19/95 Date
Single review of CCRP-declassified documents was authorized by DOE Office of Declassification memo of August 22, 1994.

	Curies	% Loss
Total Losses Accounted for	8508	21.11

Product

PE-2 (Heterogeneous sample)
Estimated Product Shipped 18500 — 45.91%
(Based on ion chamber readings)

Material Balance 27008 — 67.02%

cc: E.I. Wyatt
K.J. Witkowski
Central Files
F.L. Steahly
R.E. Blanco

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POST OFFICE BOX P
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CENTRAL FILE NUMBER
52-10-11

DATE: October 2, 1952

COPY NO. 2a

SUBJECT: Analytical Data - Run #51

TO: M. E. Ramsey

FROM: E. I. Wyatt

INV. 64

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2 6 0 a

To: M.E. Ramsey
From: E.I. Wyatt

Analytical Data
Run #51
LST 1550 9-30-52
Shipment #57

10-2-52

Batch	Curies	Slugs	A-8	% Loss
A	8070	18.48	36	0.09
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C	7721	20.42	159	0.39
D	6519	18.60	107	0.27
E	5596	17.54	133	0.33
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	40297	106.34	1089	2.70

Other Cell A Losses

	Curies	% Loss
SWC	4063	10.08
SWCW	1308	3.25
A-9 and PF Rinse	662	1.64
Total Cell A Losses	7122	17.67

Extraction Cell Losses

	Curies	% Loss
A8-Cubicle Wastes	505	1.25
Feed Tank Rinse	14	0.04
HCl Waste (MWHClE)	19	0.05
SWFN	445	1.10
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Total Losses Accounted for 8508 21.11

Product

PE-2 (Heterogeneous sample)
Estimated Product Shipped 18500 45.91%
(Based on ion chamber readings)

Material Balance 27008 67.02%

Classification changed to
UNCLASSIFIED
(insert appropriate classification level and category)
by authority of T. F. DAVIS 10-18-93
(Authority for change in classification) (Date)
by C. C. Peters 3/29/94
(Signature of Person making change) (Date)
Verified by: V. L. Norman 3/29/94
(Signature of person verifying change) (Date)

cc: E.I. Wyatt
E.J. Witkowski
Central Files
F.L. Steahly
R.E. Blanco

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CENTRAL FILES NUMBER
58-6-32

DATE: June 2, 1953

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SUBJECT: Analytical Data - Run #52

TO: M. E. Ramsey

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To: M. E. Ramsey
From: E. I. Wyatt

Analytical Data
Run #52
LST 0530-5-28-53
Shipment No. 58

Batch	Curies	Slugs	A-8	% Loss
A	10900	18.12	63	0.15
B	9600	18.89	75	0.22
C	8379	16.65	52	0.15
D	6714	15.23	92	0.26
Heels (not included)	1862	4.27	—	—
	34784	73.16	282	0.81

Other Cell A Losses	Curies	% Loss
SWO	3570	10.26
SWCW	2657	7.64
A9 & PF Rinse	272	0.78
A9 & PF acid rinse composite	6938	19.95
Feed Tank Rinse	23	0.07
Cell A Sump	164	0.47
Total Cell A Losses	13906	39.98

Extraction Cell Losses	Curies	% Loss
[illegible] (Effluent)	1	—
A-8 Resin Cubicle Waste	[illegible]	[illegible]
[illegible]	2369	[illegible]
[illegible]	566	[illegible]
PK Rinse	380	[illegible]
Total Extraction Cell Losses	3344	[illegible]

	Curies	%
Total Losses Accounted for	17250	49.59%

Product	Curies	%
PK-2	14252	40.97%
Product before drying (PK-2 minus PK Rinse)	13872	39.88%
Estimated Product shipped (Radiation)	7000	20.12%
Material Balance	31122/34784	89.47%

cc: E. I. Wyatt
[illegible] J. Witkowski
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CENTRAL FILES NUMBER
58-6-33

DATE: June 3, 1953

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SUBJECT: Analytical Data Run #52A

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A-

To: M. E. Ramsey
From: E. I. Wyatt

Analytical Data
Run #52A (Recovery)
LST 1300-6-2-53
Shipment No. 59

6-3-53

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This run was made in an effort to find the losses unaccounted for in Run #52 as well as the large amount of material held up by the filter.

Starting Material	Curies
Unaccounted for loss from Run #52	2755
AF and FF Composite acid rinses	5217
1300 [illegible]	1400
[illegible] from Run #52	2685
[illegible] from Run #52	1998
Feed Tank Rinse	17
Total Starting Material	14072

Losses	Curies	Per cent Loss
[illegible] Recovery	[illegible]	1.21
[illegible]	[illegible]	0.33
AF-FF Rinse	[illegible]	1.06
A-B Resin Cubicle Waste	[illegible]	0.22
Feed Tank Rinse	201	[illegible]
[illegible] (Effluent)	106	0.7[illegible]
[illegible]	[illegible]	[illegible]
FR [illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
Total Losses Accounted for	247[illegible]	[illegible]

[illegible]

[illegible]	112	
[illegible] (Product 52A plus [illegible] Rinse [illegible])	3654	
[illegible] from [illegible] #52	3515	[illegible].94
Product before drying ([illegible])	3606	
[illegible] Product Shipped ([illegible])	3700	

Material Balance [illegible]

cc: E. I. Wyatt
[illegible] Mikowski
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CENTRAL FILES NUMBER
58-7- 77

COPY NO.

DATE: July 14, 1953

SUBJECT: Runs 53 and 53A, Shipment Nos. 60 & 61

TO: M. E. Ramsey

FROM: E. I. Wyatt

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To: M. E. Ramsey
From: E. I. Wyatt

Analytical Data
Run No. 53
LST 0620, 7-7-53
Shipment No. 60

Batch	Curies	Slugs	A-8	% Loss
A	11446	17.1	50	0.07
B	11110	17.1	101	0.15
C	10241	17.0	114	0.17
D	11041	20.3	230	0.34

Metathesis Waste*	1819
Acetate Feed	38375
Versene Feed	2629

Resin Cubicle Losses	Curies	
A-8 Resin Cubicle Waste	783	1.16
HCl Effluent	40	0.06
MW $NaNO_3$	56	0.08
MW Fuming Nitric*	3310	—
Resin Wash	463	0.68
Titration HNO_3	6	0.01
Total Resin Cubicle Losses	1348	1.99

PE-2	20000
PE-Rinse*	3068

Product shipped, radiochemical (Note a), 16,932 curies

Note a. The fuming nitric acid separation of Ba from La was incomplete and the radiation readings of the product cone were not valid.

* Samples with asterisk salvaged and combined with Run No. 53A

No material balance could be made on Run 53 or 53A separately because no rinse of the vessels was made until Run 53A had been processed. Run 53A was therefore combined with a part of the material unaccounted for in Run 53. The combined material balance shown on page 2 gives a more accurate picture than separate material balances would do even though two separate shipping times are involved.

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Run No. 53A
LST 0915, 7-9-53
Shipment No. 61
And Material Balance of Runs 53 and 53A

Batch	Curies	Slugs	Curies A-6	% Loss
E	7495	16.1	220	0.32
F	9229	20.3	112	0.17
G	7198	16.8	92	0.14
Total (No. 53 and 53A)	67760	124.7	919	1.36%

Other Cell A Losses (Run 53A)	Curies		%
SWR Metathesis Recovery	294	734	0.43
A-9 and Process Filter Rinse	955		1.41
Feed Tank Rinse	1887		2.78
Total Cell A Losses (Runs 53 and 53A)	4055		5.98%

Resin Cubicle Product	Curies
Acetate Feed	1320
Versene Feed	31000
	32320

Resin Cubicle Losses (Run 53A)	Curies	%
Resin Cubicle Waste	292 ✓	0.43
MWHCl Effluent	88 ✓	0.13
MW Fuming Nitric	821 ✓	1.21
MW $NaNO_3$	295 ✓	0.44
F.R. Rinse	1830	2.70
Total Resin Cubicle Losses (53A)	3326	4.91
Total Resin Cubicle Losses (53 & 53A)	4674	6.90
Total Losses Accounted for (53 and 53A)	8729	[illegible]

Product	Curies	%
PE-2 (53A)	35632	———
Estimated Product Shipped (53A)	33802	———
Total Product (53 and 53A)	50734	74.87
Material Balance		87.75

35632
1830
33802

cc: M. I. Wyatt
R. J. Witkowski
R. E. Blanco
Central Files

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OAK RIDGE, TENNESSEE

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CENTRAL FILES NUMBER
53-12-19

re: RUN 54

DATE: December 3, 1953

COPY NO. 2 A

SUBJECT: Summary of Run No. 54 With Significant Analytical Results.

INV. 64

TO: A. F. Rupp

FROM: E. J. Witkowski

Distribution

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2. A. F. Rupp
3. F. L. Culler
4. E. I. Wyatt
5. E. J. Witkowski
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[redacted]

Summary of Run No. 54 With Significant Analytical Results

To: A. F. Rupp

From: E. J. Witkowski

December 3, 1953

The run was started on November 4, 1953, with the loading of 184-4" and 90-8" Hanford slugs containing approximately 102,000 curies of product. Before the run was started, a decision was made to use filter aid in the extraction and metathesis filtrations if the process filters became seriously plugged as had normally occurred in most previous runs. The same plans for using filter aid had been made before Run No. 53 was begun. However, the process filter operation was unusually successful during that run and the filter aid was never used.

The original plans called for splitting the run into two or three batches through the resin column in order to maintain the activity on the column below its capacity. The first three batches were to constitute the first portion of the run.

The sulfate extractions of batches A and B were filtered but with great difficulty because of plugged process filters; it was necessary to leave heels of approximately 15 gallons because no filtration progress could be made past that point.

Only a very small portion of batch C was filtered before the process filters plugged completely. It was at this point that filter aid was added to the extraction slurry. It did not improve the filtration, however, and the separation had to be made by the old decantation method. The extraction separation losses, in spite of the filtration difficulties, were relatively low as indicated in the following table. (All analytical results in this report calculated to 8:00 A.M. on November 24).

Batch	Curies In Dissolver	Extraction Loss-Curies	
A	5,264	29	(filtered)
B	5,221	42	"
C	5,660	143	(filtered & decanted)
Total -	16,145	214	

The extraction water washes following the first three batches were also decanted because of plugged filters. The decantation of water washes had always been highly satisfactory in the past but in this case practically all of the product was lost (analysis showed 19,120 curies; it was obviously not very accurate because

[redacted]

[redacted]

Summary of Run No. 54 With Significant Analytical Results

the sample was a slurry). Before the analytical results on the water washes became known, a metathesis was started in the extraction tank. This metathesis was discontinued and a decision made to bypass the first three-batch portion of the run. The small quantity of product left in the extractor was then combined with the water washes and transferred for storage in the neutralizer A-5.

All tanks and filters were thoroughly cleaned before batch D extraction was begun. The UNH from batch D, which had been dissolved while the extractions of the first three batches were carried on, was then filtered through the unused crud filter and extracted. The extraction filtration was completed with difficulty but no decantation was used. The loss on this filtration was relatively high, 927 curies. The waste was settled and decanted in another tank, A-11. The loss from this decantation is not known because of lost analytical results although it can be safely assumed that it was low.

Before the dissolving of batch E was begun, 50% caustic was boiled in the dissolver to clean out any silica that may have been interfering with the process. Filter aid was used in the crud filtration of the UNH of batch E as well as batch D although none was added to the extraction tank.

The process filters plugged after 15 gallons of batch E extraction was filtered. A decision was then made to discontinue the use of the process filters until all of the material processed thus far was cleaned up by extracting and metathesizing in tank A-11 where separations by decantation were almost always successful.

The sulfate slurry of batches D and E was then transferred to tank A-11 which contained the heel from the redecanted waste from batch D. Batches D and E were then successfully redecanted with a loss of 814 curies. Additional significant analytical results up to this point follow:

Batch	Curies Dissolved	Extraction Loss-Curies 1st Separation	2nd Separation
D	5,792	927 (recovered)	<927 (actual analytical results lost)
E	4,562	422 (filter loss not recovered)	814 (not recovered)
Total A through E	26,439	2,377	

[redacted]

Summary of Run No. 54 With Significant Analytical Results

Loss in washing tanks and filters before batch D extraction - 324

Product in A-11 (calculated) - ~23,738 or 91% of product dissolved thus far

The material containing the product from batches A, B and C was then jetted to A-11 on top of the extraction heel of batches D and E. The combined product of all five batches was precipitated as a sulfate and washed successfully four times with water using the decantation separation. The losses follow:

Extraction Loss	73
Extraction washes -	380

Roughly 90% or 23,000 curies of the product from the first five batches was calculated to be in A-11 when the metathesis was started in that tank. The decanted wastes from the two metatheses, however, carried approximately 45% or 10,600 curies of this product.

The carbonate heel in A-11 was dissolved in 6M HNO_3 and transferred to the extractor through a crud filter which was precoated with filter aid. The tanks and filters were thoroughly washed before this transfer was made. The 10,600 curie metathesis decant waste was also transferred to the extractor where the carbonate concentration of the resulting slurry was calculated to be .7M K_2CO_3. An attempt was made to filter this material but only 30 gallons could be put through; this waste filtrate contained about 7,000 curies of product.

The filtrate was brought back to the extractor and the slurry adjusted to 2M K_2CO_3. While the slurry was settling in preparation for decantation, tanks A-17, A-11, A-8 and the crud filters were thoroughly cleaned to eliminate filter aid. It had become apparent by this time that the filter aid was not being separated out of a slurry by the crud filters. Additional significant analytical results to this point follow:

Loss in tank and filter rinse before transfer to extractor - 1,277
Loss in tank and filter rinse while material settled in extractor - 958

The decantation of the material from the extractor resulted in a loss of 11,962 curies out of a calculated 21,050 which were supposed to be in the extractor. The waste containing this large amount of product was discarded with no further attempt at recovery. The remaining material in the extractor was

[redacted]

Summary of Run No. 54 With Significant Analytical Results

washed and processed through the resin column cubicle.

Carbonate decantation loss	-	11,962
Water wash loss	-	292

The analysis of the two feed solutions in the resin cubicle feed tank showed a total of only 1,407 curies of product with 460 curies left in the extractor rinse because of an incomplete transfer. The feed material was processed to completion with a total resin cubicle loss of only 22 curies.

Based on the known losses to the end of this portion of the run, the amount of product taken off the column should have been 8,314 curies. The actual analysis, however, showed only 1,544 curies with no account for 6,770 curies or 25.6%. The bulk of this product was undoubtedly lost through leaks around the process filters which developed during this run.

Extractor and filter rinse following transfer to cubicle	-	460
Resin cubicle losses	-	22
Total losses accounted for	-	18,125
Product in product tank	-	1,544
Starting material from dissolver	-	26,439
Material balance	-	74.4%

Although the run, thus far, was almost a total loss, it was nevertheless educational. The poor extraction filtrations of the first three batches were first believed to be caused by incomplete removal of silicon compounds from the UNH solution because the crud filter used may possibly have had a broken seam. It also became evident during the third batch that the addition of filter aid to the extraction slurry does not speed up its filtration through the process filter.

The filtration of the fourth and fifth batches was made through the spare crud filter, not used up to that point; the filtration was not improved. It was then concluded that both crud filters may have had the pores enlarged by previous decontaminations and cleaning.

After all five batches were extracted and metathesized in tank A-11 and filtered through a crud filter to the extractor, a sample of the material indicated that the solution contained a large amount of filter aid. At this point it became obvious that the crud filters were not only ineffective in removing the silicon

[redacted]

compounds, they were also ineffective in retaining the recommended Celite #545 filter aid.

Tests have since been run on spare crud filters which have never been used. It was found that even the new crud filters passed an appreciable quantity of the Celite #545 filter aid.

The evidence gathered in the first part of this run may also explain the successful process filtration in Run No. 53, the most successful run to date. The pores in the UNH crud filters were undoubtedly very small during that run as evidenced by the excessive time required to filter each UNH batch. The resulting thorough clean-up of the UNH was probably very effective in easing the load on the process filters.

The second portion of the run, batches F, G, H and I, was also processed to completion with practically no success. The bulk of the product appeared to have been lost through leaking equipment in the process filter cubicle and in wastes coming through the process filters.

No filter aid was used in the UNH crud filtration of any of the last four batches; the UNH filtered through with no difficulty. All extraction slurries, with the exception of the last three gallons of the last batch, were decanted twice to reduce losses. The heel of the last redecanted batch was washed back to the extractor and filtered with the three gallon heel. The water washes following the extractions were filtered, with difficulty.

Batch	Curies In Dissolver	Extraction Loss-Curies
F	4,941	43
G	3,980	509
H	3,718	152
I	2,996	47
Total	15,635	751

Water wash loss - 110

The metatheses and the metatheses washes were filtered but the combined loss was extremely high, 6,804 curies. This was the highest loss ever encountered in a filtered waste and there is no apparent explanation for its occurrence. This waste was temporarily bypassed and the balance of the product processed through the resin cubicle.

Summary of Run No. 54 With
Significant Analytical Results

With the known losses encountered thus far, the amount of product entering the resin cubicle feed tank was calculated to be 7,970 curies. The analyses of the feed however, accounted for only 1,983 curies. The balance was undoubtedly again lost through the leaking equipment in the process filter cubicle. The product entering the product tank to be combined with the batch from the first portion of the run was calculated to be 1,732 curies.

Loss in Metatheses and Metatheses Washes	-	6,804
Total losses accounted for	-	7,665
Product in resin cubicle feed tank	-	1,983
Starting material in dissolver	-	15,635
Material balance to resin cubicle feed tank	-	55.3%

Again, the amount of product was too small for shipment so the metatheses waste containing 6,804 curies, was again processed through the sulfate and metatheses steps as a third portion of the run. The losses through the filter however, were again too high to make an adequate amount of product for shipment so the run was discontinued.

Extraction waste	-	1,087
Metatheses and washes	-	3,464
Extractor rinse following transfer	-	146
Total loss		4,697
Product in cubicle feed	-	428
Starting material	-	6,804
Material balance	-	75.4%

Decontamination of both filter cubicles, in preparation for repairs, was started immediately following the run. Work was also begun on a series of tests to determine the effectiveness of the present coating removal and filtration procedures. It is believed that enough information can be gathered from these tests to prevent a repetition of the operating difficulties encountered during this run.

E. J. Witkowski

EJW:hg
cc: M. E. Ramsey
A. F. Rupp
F. L. Culler
E. I. Wyatt
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RUNS 55-58

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RALA PRODUCTION – 1954

A. F. Rupp and E. J. Witkowski

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For The Atomic Energy Commission
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OPERATIONS DIVISION

RALA PRODUCTION – 1954

A. F. Rupp and E. J. Witkowski

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RaLa PRODUCTION – 1954

A. F. Rupp and E. J. Witkowski

PRODUCTION

Approximately 88,000 curies of RaLa product was produced in three runs and shipped in four batches. Except for the period from April 29 to July 16, when a run was completely lost and operations were shut down because of contamination following an accidental release of activity, production schedules met the demands of Los Alamos.

As in previous years since the ion-exchange method of final separation and purification was adopted, the product contained impurities which necessitated some additional processing at Los Alamos before it could be used. This condition inconvenienced the Los Alamos personnel but did not affect their experiments, since the quantities contained in the shipments were sufficiently large to compensate for the losses incurred by the additional processing. The impurities appeared to be organic material resulting from radiation and chemical breakdown of the ion-exchange resin.

The process yields (see Table 1) continued to be low in 1954; the total product shipped represented only 53.4% of the total starting material (excluding decay). The following five steps in the process accounted for practically all the known losses.

Dissolver Heels. Product was left behind in the dissolver because the quantity was too small to justify the time required for its recovery.

Metathesis Filtration. A high loss was experienced only in run No. 58. It may have been caused by any one of three changes in the metathesis operation or by a combination of them: (1) a reduction in the amount of inactive barium caused by a very short irradiation time in the reactor; (2) a reduction in the amount of lead in order to speed up filtration; (3) an increase in the

TABLE 1. ANALYTICAL SUMMARY

	Amount of Activity (curies)			Total Activity	
	In Run 55	In Run 57	In Run 58	(curies)	(%)
Dissolver starting material	84,683	37,020	42,664	164,367	100.0
Cell A losses					
Dissolver heels	*	2,156	5,498	7,654	4.7
Sulfate extraction filtrations	872	171	308	1,351	0.8
Metatheses filtrations	534	198	7,841	8,573	5.2
Extractor and filter rinse	186	4,892	2,000**	7,078	4.3
Total cell A loss	1,592	7,417	15,647	24,656	15.0
Resin cubicle losses					
Fuming nitric filtration	8,152	655	1,839	10,646	6.5
Product evaporator rinse	2,930	2,550	4,814	10,294	6.2
All other	444	191	189	824	0.5
Total cubicle loss	11,526	3,396	6,842	21,764	13.2
Total of all known losses	13,118	10,813	22,489	46,420	28.2
Product shipped (Los Alamos measurement)	45,091	22,800	19,942	87,833	53.4
Material balance	68.7%	91.1%	99.5%	81.7%	

*The slug heels were used in a test run.

**Loss was estimated because of sampling difficulties.

volume of K_2CO_3 solution from 9 to 50 liters in an effort to avoid the incomplete reactions experienced in some previous runs.

Extractor and Filter Rinse. Product was left behind in the extractor tank and on the process filters after it was transferred from the extractor to the resin column feed tank. It is impossible to make complete transfers because the relatively small size of the feed tank limits the volume of solutions that can be used for cleaning out the large extractor tank.

Fuming Nitric Acid Filtration. The highest loss experienced in this step was incurred in run No. 55, the only run processed through cubicle No. 300; this cubicle has a product evaporator and filter of the old design, and it is believed that the high loss was caused by cracks in the filter resulting from fabrication difficulties.

Product Evaporator Rinse. Some product was left behind in the evaporator after the main portion of the product solution had been transferred to the shipping cone. The volume of water used for the dissolution and transfer of the product is limited by the size of the cone and is too small for an effective transfer. Attempts to wash the tank with more water and to transfer the water to the shipping cone after the first portion had evaporated resulted in higher losses from the cone into the off-gas line.

In addition to the losses determined by the analysis of waste solutions, it is estimated that more than 15% of the starting product was lost through the off-gas line during the drying operation in the shipping cone. The largest single loss occurred in run No. 55. Since ORNL does not have the facility for making a radiochemical analysis of the product after it is put in the drying cone, such an analysis was made at Los Alamos; their analysis was lower by 20,000 curies than the measurement made at ORNL after the fuming nitric acid step.

UNUSUAL INCIDENTS

The equipment was operated far above its designed capacity (500 curies in dissolver and extraction section and 10,000 curies in the purification section) in every run. This condition led to the most serious accidental release of activity ever experienced in the history of the process, which made it necessary to abandon a run and to shut down the building for decontamination for a period of 11 weeks.

The slugs loaded into the dissolver for this run contained approximately 100,000 curies of product. The quantity of starting material was unusually large so that a very large shipment could be produced and so that the chance of a poor shipment resulting from losses caused by the usual processing difficulties could be reduced. It was planned to make the run in two parts and two separate shipments.

Dissolvings for the first part were discontinued after the third batch, instead of after the fourth batch as had been planned, because the UNH analysis showed that the slugs contained more than the expected amount of activity. The partially dissolved uranium slugs left in the dissolver therefore were hotter than in any previous run. The incident occurred at the beginning of the addition of 60% HNO_3 for the second part of the run. Because of their intense radioactivity, the slugs apparently had become very (thermally) hot during the period of time that they were not covered with liquid between the dissolvings for the two parts of the run and had reacted violently with the HNO_3. The solution containing the radioactivity was blown into the operating area through the slug chute and the solution addition lines.

Following the incident, the radiation levels in the RaLa building itself were too high to permit entrance for effective decontamination. Work was started after the short-lived activity was allowed to decay for a period of ten days. Several other Laboratory areas north of the RaLa building were also contaminated, principally with short-lived iodine. Fortunately, the incident occurred during the 4–12 shift, when these areas were not occupied; by the use of emergency crews, most of the contamination was cleaned up before personnel reported for work at 8:00 AM the next day.

The incident caused a great deal of inconvenience in Laboratory operations and some lost time, but there was no serious overexposure of Laboratory personnel. The operators who were in the most vulnerable position at the time of the accident were quick to recognize the hazard and to protect themselves, being well practiced because of the frequent use of the RaLa emergency procedures.

As a result of this incident and of the frequent hazards encountered in the production of the large batches of RaLa, Los Alamos was requested to review its requirements and to reduce the number and size of the batches requested, if possible. An agreement was reached that the Laboratory will attempt no more double runs and that only four runs per year will be made. It is believed that the new schedule will substantially reduce the operating hazards.

Direct pipe connections between the process vessels in the cells and the operating area (which are considered by the Operations Division to be obsolete design) were responsible for several other backups of activity into the operating area. The most serious of these occurred during run No. 55, in January, when activity backed up to the operating panel board through a steam line servicing a jet in cell A. While an attempt was being made to decontaminate the line with steam, a small valve at the steam pressure gage failed and activity was blown into the operating area. The building was contaminated, and two operators received some radiation overexposure – fortunately, not a serious amount.

General air contamination that was experienced through a large portion of the Laboratory on three occasions during two of the three successful runs was attributed to the RaLa operations; however, there is a good possibility that some of this air activity came from the experimental reactor area. The condition was presumably caused by a high discharge from the stack; in one case the caustic line to the dissolver off-gas scrubber plugged, and in another case, during an atmospheric inversion, the scrubbers operated normally but not effectively enough.

EQUIPMENT

The equipment used for the production of RaLa has always been operated far above its design capacity; changes to allow for higher levels of production were made only in the final purification stages, and they were inadequate for very large batches. The last alterations in the final purification equipment were made in order to produce 10,000-curie batches; however, before the equipment was completed, a new goal of 30,000 curies was set, and 50,000-curie batches were actually produced. Operation at these very high radiation levels has materially increased the hazards, and the equipment has deteriorated over a period of years.

It was decided several years ago that it would not be practical to further alter the equipment to increase the capacity, and a proposal to build new, adequate facilities was submitted to the Atomic Energy Commission. However, it was decided this year to build the new plant at Arco; the new plant is expected to be in operation by July 1, 1956, and the existing RaLa plant will be permanently shut down at that time.

Maintenance of the equipment has continued to be difficult and expensive. For long-term operations, it would be more practical to replace or to rebuild some parts than to make the excessive repairs that would be needed. However, since the present plans are to produce only six more batches, no replacement of equipment or rebuilding will be done except where absolutely necessary.

The only major maintenance job undertaken this year was to rebuild one of the cubicles which hold the ion-exchange equipment. This was necessary because of a leak in the product evaporator thermocouple well and the failure of the sampling valves. The evaporator was replaced by one of a greater capacity and of an improved design that permits much closer control of the evaporation operation.

The RaLa equipment at the present time is in only fair condition. The cell A transfer lines are known to have many leaks, and the thermocouples in the dissolver have been inoperable for years. Decontamination and repairs in this cell are inadvisable and will not be done unless some major piece of equipment fails completely. The high level of contamination on the concrete walls and floor would require the removal of a large quantity of highly radioactive concrete, and it would be impossible to complete the work fast enough to meet the Los Alamos schedule. Also, a major decontamination job in this cell would be very expensive and would require a great deal of radiation exposure to operating and maintenance personnel. It is hoped that this work can be avoided, since RaLa operations at the Laboratory are to be permanently discontinued.

The main operating difficulties with the purification equipment are the erratic operation and the frequent failure of the sampling plug valves; both conditions are caused by the deterioration of the Teflon seats.

It is believed that one more decontamination of one of the cubicles will be required to complete the last six runs scheduled at the Laboratory.

PROCESS IMPROVEMENTS

Several important changes were made in the process this year. One was the addition (to the standard procedure) of a second fuming nitric acid precipitation after the nitric acid elution of the product from the ion-exchange column. Prior to the last run of this year, the second precipitation was made only when the transfer of product from the evaporator to the cone was incomplete. The procedure was standardized after Los Alamos had reported that run No. 57 contained some product in an insoluble form. Since only one fuming nitric precipitation was used in that run, it was thought that the barium sulfate precipitate, formed by the breakdown of the sulfonated vinyl ion-exchange resin, was not completely converted to a nitrate. The insoluble material was apparently eliminated in the last run, No. 58.

As previously mentioned, a very large portion of one run was lost into the off-gas line during the transfer of the product solution into the shipping cone and the drying operations. Also, smaller but substantial amounts of product were lost during the same operations during runs made in previous years. The losses were caused by the high level of liquid in the shipping cone during the drying operation (because of the size of the cone) and by the type of drying operation, which involves drawing hot air over the surface of the product solution without heating the cone itself. It is apparent that this method is quite ineffective; the evaporation of the product is almost completely dependent upon the heat generated by the radiation energy of the product itself, as well as upon some heat that is transferred to the solution by conduction from the charging head, through the top of the cone.

Since it was impractical to revise the equipment, a series of drying tests was run in an attempt to change the operating procedures to compensate for the inadequacy of the equipment. It was found that the losses could be reduced to a minimum by lowering the temperature and amount of air passed over the cone; also, it was shown that a large amount of product could be lost by transferring product solution into a cone that contains dried product. It was apparent that it would often be more preferable to lose some product by an incomplete transfer from the evaporator to the cone than to add an evaporator rinse to the cone after a portion of the product had dried.

After much difficulty was experienced with incomplete metathesis during run No. 57, the process was revised by increasing the amount of K_2CO_3 reagent in one metathesis from 9 to 50 liters. This change in standard procedure also had been made in a previous year because of similar difficulties but had soon been discontinued because of the difficulty in filtering the larger volume. More recent changes made to alleviate filtration troubles now permit the use of larger volumes.

The volume of solution used to transfer the product from the extractor to the resin column feed tank was also increased in run No. 58 in order to accomplish a more complete transfer. The volume of HNO_3 used for the dissolution of the carbonate cake prior to transfer was increased, by dilution, from 10 to 18 liters. This change was made possible by the replacement of the original feed tank with one of a larger size when the cubicles were rebuilt.

One of the most difficult parts of the process in previous years was the filtration of the lead-barium slurries after the sulfate precipitation in UNH and after carbonate metathesis. A complete run was lost in November 1953 as a result of these difficulties; it became evident during this run that plugging of the process filters was caused by fines from the Celite filter aid used in the preceding UNH "crud" filtration operation.

Following the loss of the run, this part of the process was examined to determine whether the use of a filter aid could be discontinued or whether the quality of the filter aid could be improved if it were found to be essential. A memorandum was issued on this work and is appended to this report.

Further tests showed that the procedure for removing the aluminum slug jacket was not effective in removing the silica slug-bonding material and that by changing the procedure it would be possible to remove the silica from the process before filtration and therefore eliminate the need for a filter aid. Following the laboratory investigation, a full-size, single-batch-test run was made with the new procedure and was found to be highly satisfactory. The procedure was used in the four production runs made this year, and only minor filtration difficulties were encountered.

FUTURE PLANS

All plans for the future production of RaLa at ORNL are based on the Atomic Energy Commission's plans to relieve the Laboratory of the RaLa production work in July 1956. As previously mentioned, the only repairs that will be made are those that are absolutely necessary to fulfill the

Laboratory's commitment for RaLa deliveries up to that date. The requirements for manpower are also planned only to that date. Some manpower, lost as a result of normal turnover, has not been replaced because of the expected loss of the RaLa project; the remaining personnel will be used to operate the new Fission-Product Pilot Plant, the operation of which is expected to start at about the same time that RaLa operations are discontinued.

APPENDIX

INTRODUCTION

Since the major equipment and process revisions were made almost three years ago, RaLa operation has been hampered by the frequent plugging of the process filters during the filtration of lead-barium sulfate precipitate out of UNH and the filtration of lead-barium carbonate slurry after metathesis. Run No. 54 was completely lost in November, 1953, as a result of this difficulty. It became evident during this run that the process filters were being plugged by an incomplete removal of foreign material, probably silica, from the UNH in the preceding crud filtration step and that the fines in the Celite 545 filter aid were passing through the crud filter and interfering in the subsequent filtrations through the process filters (a complete report on run No. 54 is given elsewhere[1]). Therefore it was decided to re-examine certain parts of the process.

The investigation was carried out in two parts: a check was made of the effectiveness of the coating removal procedure in removing the aluminum and the silicon alloy bonding material, and an investigation of the filter aid was made in order to determine whether it could be improved or possibly eliminated. The coating removal tests and filter aid classification were performed by P. B. Orr of the Operations Division. The microscopic studies and particle-size determinations were made by T. E. Willmarth of the Analytical Chemistry Division.

COATING REMOVAL

The effectiveness of the coating removal procedure was visually ascertained by means of a test which roughly duplicated, on a small scale, the usual procedure. The coating and bonding removal was performed on two 4-in. Hanford slugs in a stainless steel bucket heated with a gas burner.

[1]E. J. Witkowski, *Summary of Run No. 54 with Significant Analytical Results*, ORNL CF-53-12-19 (Dec. 3, 1953).

First, the aluminum was removed by a solution of 10% NaOH and 20% $NaNO_3$ at a temperature of 100 to 105°C for 1 hr; this step was then repeated in order to ensure complete removal of the aluminum. It was apparent that the removal of the aluminum was complete after these two steps and that the second may have been superfluous. A black-looking bonding material, however, was left on the uranium metal and had been practically untouched.

The two slugs were then treated with 44 lb of 6% HNO_3 containing 6.4 g of $Hg(NO_3)_2$ at a temperature of 100°C for 3 hr. This step, which was supposed to have undercut the silicon alloy bonding, removed only a very small portion of the material, which readily settled to the bottom of the dissolver as a black, flaky precipitate. The bulk of the bonding material remained on the slugs. The unremoved bonding material would normally stay in the UNH as a precipitate after the uranium was dissolved and, as tests later showed, would plug the UNH crud filter unless filter aid were used.

The procedure for undercutting the bonding was revised: the slugs were heated at 100°C in a 20% HNO_3 solution for 34 min. The black bonding material which had settled out in the bottom of the dissolver as a flaky precipitate was completely removed. The surface of the slugs became bright and clean and showed little damage from the action of the acid. The solution contained only 4.94 mg of uranium per milliliter of solution; this loss of uranium would be considered insignificant in the RaLa process. The solution also contained 4.6 ppm of silicon.

In order to determine the extent of treatment with 20% HNO_3 that would be required, the slugs were again heated in 20% HNO_3 for 15 min. There was no apparent precipitate in the solution, and it contained 3.0 mg of uranium per milliliter and 3.4 ppm of silicon; the proportion of silicon to uranium was roughly the same in both cases.

An attempt was then made to find a suitable method for dissolving the fine pieces of bonding alloy deposited on the bottom of the dissolver so that their removal would be facilitated. In order to get an adequate amount of this fine alloy deposit for test purposes, ten ORNL slugs were dejacketed, and the bonding was removed by means of the procedure used for the first two slugs.

The only solvents found which would dissolve the bonding material in a reasonable period of time were solutions containing free HF. Cold solutions of 5% NH_4HF_2 and NH_4F dissolved the precipitate rapidly but reacted on the uranium slugs and formed another precipitate, which was identified as UF_4. The formation of the UF_4 precipitate plus the probable long-term damage to the equipment by the corrosiveness of the fluoride chemicals made their use unattractive in the RaLa process.

Solutions of NaOH and KOH up to 50% concentrations, with alternate treatments with HNO_3 up to 70% concentrations, showed no visible effects on the bonding alloy, even after prolonged periods of boiling in these solutions.

It was apparent that the removal of the bonding material from the dissolver would have to be accomplished by mechanical means, that is, vigorous washing. Since the bonding material is relatively heavy and not easily suspended by agitation with water, a decision was made to try 50% NaOH.

To test the effectiveness of the 50% NaOH in suspending the bonding precipitate, eight more 4-in. Hanford slugs were dejacketed, and the bonding was undercut with 20% HNO_3. The dissolver was emptied except for the slugs and the bonding precipitate, and, after 50% caustic had been added, the mixture was boiled and sparged. The caustic was then syphoned out and was observed to carry a large portion of the undissolved precipitate.

FILTER AID TESTS

As mentioned in the introduction, there was evidence that some filter aid passed through the UNH crud filter and contributed to the difficulties experienced in run No. 54. After the run was completed, the crud filters (porosity G Micrometallic stainless steel) were decontaminated, removed, and tested. A water slurry of filter aid was filtered, and the filtrate was observed to be slightly cloudy. When the filtrate was boiled down, a white precipitate settled out and was identified under a microscope as filter aid. The same test, made on two replacement filters, gave identical results.

Literature by the Micro Metallic Corp., the manufacturer of the filter used, indicates that porosity G filters have a mean pore opening of 10 μ and that they can be expected to remove particles down to one third the diameter of the mean pore opening. Since the microscopic examination of the Celite 545 filter aid revealed that 57% of the particles were under 4 μ in diameter and that many of the particles were less than 1 μ in diameter, it is not surprising that some of the filter aid sifted through the filter.

The penetration of the filter by some small particles of filter aid might present no problem if it were possible to precoat the filter and to wash it with an adequate amount of water to eliminate the small particles. The filter might also work satisfactorily if it were possible to recirculate the UNH through the filter. Unfortunately, neither of these procedures is possible. The UNH must be added to the water from the precoating operation, and it cannot be recirculated because of the nature of the equipment.

In order to roughly check the possibility of eliminating the filter aid in performing the UNH filtration or of improving it should its use prove to be necessary, three, two-slug batches of UNH were made by dissolving the slugs which had been thoroughly cleaned during the testing of the coating removal procedures. It was, of course, impossible to duplicate the exact operating conditions by doing the dissolving operations in a stainless steel bucket. The time required for the dissolving operation was much more than that normally encountered in the regular RaLa process, and it was necessary to make additions of HNO_3 while the dissolvings were in progress in order to compensate for the acid which evaporated.

It was found that all batches could be filtered (although the rates varied considerably) through a small laboratory-type, stainless steel, porosity G filter if air was periodically blown back through the filter when the rate dropped below a practical level. This experiment could not be considered as conclusive because the filtration rates varied between batches and because the crude equipment used made it impossible to approach the true operating conditions in the RaLa process. The experiment served to indicate that filtration without filter aid was possible and that it might be a practical process in the RaLa equipment.

Since filtration without the use of a filter aid might not prove to be possible, an attempt was made to improve the grade of filter aid. A 3-in.-dia × 6-ft-long glass tube with a glass filter on the bottom and provision for overflow on the top was used to hydraulically classify the Celite 545 filter aid for the next RaLa run, and enough filter aid was classified for the next complete run. An examination of the classified material under an electron microscope showed that the 57% of the fines in the Celite which were under 4 μ in diameter could be reduced by this crude method to 34% (see Figs. 1 and 2). This material, although not completely satisfactory, would be an improvement over that used in all previous runs.

A small laboratory-type stainless steel filter was precoated with the classified filter aid and was used to filter some of the UNH solution. The filtration was satisfactory, with no noticeable decrease in rate. A second filtration of the filtrate was made without the use of filter aid, with no appreciable change in rate.

In addition to trying to improve the quality of the normally used diatomaceous-earth filter aid, a relatively new type of carbon filter aid, called "Nerofil" (produced by Great Lakes Carbon Corp.), was tested. The rate of filtration through a precoat of this material was only about one third as rapid as that of filtration through diatomaceous earth, but the flow was steady. There is a possibility that this filter aid might be more satisfactory than the siliceous type because it would be inert to all the chemicals used in the RaLa process.

EXPERIENCE WITH AN EXPERIMENTAL RUN AND A PRODUCTION RUN

Based on the results obtained in these tests it was decided to make a RaLa run without the use of a filter aid in the filtration processes. An air line was connected to blow back the UNH crud filters to help clear them whenever they plugged. In the event it became necessary to use filter aid, the classified Celite material would be used.

To improve the chances for a successful filtration, with or without filter aid, the coating removal procedure was revised to include a 20% HNO_3 undercutting of the bonding material, the water washes were increased in volume and number, and the washing techniques were revised to flush out the precipitated bonding material.

A decision was also made to test the Nerofil filter aid on a full-scale batch of UNH if some slugs were left over after the next RaLa run.

The new slug coating and bonding removal and the UNH filtration procedures were tried on a full scale, first in a 138-curie experimental run and then in a 65,000-curie production run (No. 55).

The experimental run was made with four batches of UNH dissolved from a mixture of twelve 4-in. W slugs, fifteen 8-in. W slugs, and 130 X bonded slugs. The UNH filtration of all four batches was successfully accomplished without the use of filter aid, at an average rate of 15 min per batch, or 3 gpm; it was not necessary to stop the filtration to unplug the filters at any time. The sulfate extraction filtrations averaged approximately 40 min per batch, or 2.25 gpm. The two metatheses, with a combined volume of 30 gal, filtered in 82 min, or at an average rate of 0.37 gpm. The filtration rates, as a whole, were the fastest and the most consistent ever experienced. At no time was it necessary to delay processing because of an excessive time required for filtration.

The production run which followed was made from 192 W slugs dissolved in ten batches. It was the biggest and the most successful run to date. The same new procedures were followed as in the test run. The results were comparable except for the necessity of periodically speeding up the UNH filtration by blowing back air through the filters. The first batch of UNH seriously plugged one of the filters, but at no time was processing delayed by filtration difficulties. The plugged filter was easily cleaned after the run was over by using 20% NaOH. No difficulties were experienced with the sulfate extractions and metatheses filtrations.

The Nerofil filter aid was tested in the filtration of a batch of UNH left over from the production run. Only 4.5 slugs of uranium were found to have dissolved, as compared with 19 in a normal batch. The UNH filtration rate was only 1 gpm, as compared with an average of 3 gpm in the test run without filter aid, but the rate was consistent and it was not necessary to blow back the filter.

Fig. 1. Unclassified Filter Aid. 500X.

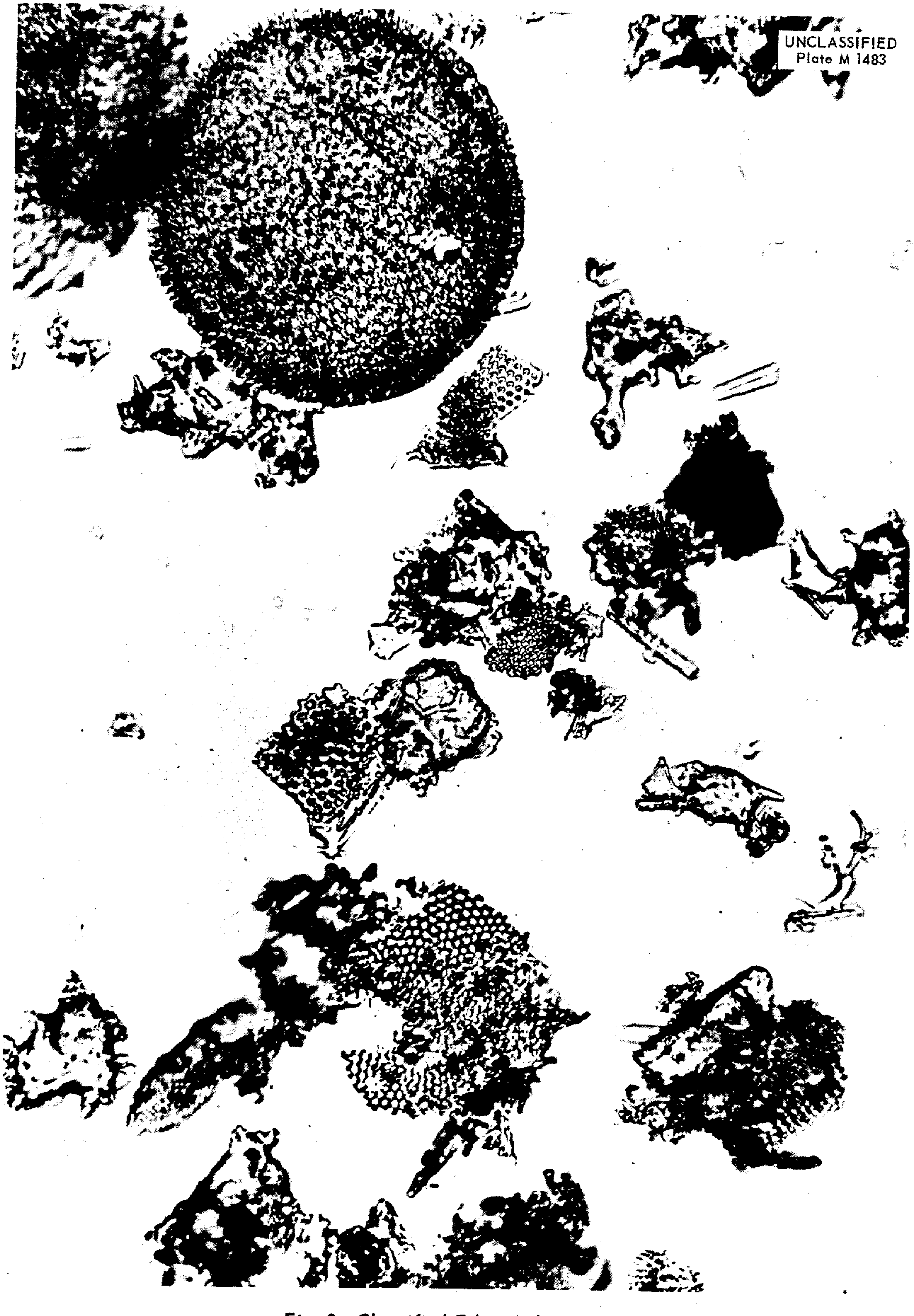

Fig. 2. Classified Filter Aid. 500X.

The rate was approximately that expected. The metatheses and extraction filtrations were comparable to those in the experimental run and production run No. 55. The total activity contained in this batch was 872 curies. All analyzed waste losses were low, but 158 curies (18%) could not be accounted for in the material balance. There is a remote possibility that the unaccounted-for loss was a result of using the Nerofil filter aid.

CONCLUSION

Because of the success of the production run, which can be at least partially attributed to the elimination of filter aid in the filtration of UNH, a decision was made to follow the same procedure in all future runs. In the event that filtration without the use of a filter aid becomes difficult, Nerofil will be tried in place of the Celite 545 previously used.

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The run was started on July 16, 1954 and successfully completed on July 23, 1954 with a shipment of 22,800 curies based on Los Alamos' measurements. Our own direct radiation measurements were unreliable because the product consisted of two parts each having a different "last separation" time. The radiochemical analyses of the product were also unreliable because of a sulfate precipitate in the product solution and because of mechanical difficulties experienced in operating the sampling equipment which undoubtedly resulted in a poor sample. Los Alamos estimated that approximately 10% of the product was in the sulfate form.

The metathesis step of the procedure again caused difficulties. Only about 30% of the total product could be dissolved and transferred from the extraction tank to the resin column cubicle after each double metathesis. It was necessary to split the run and to process it through the resin cubicle in three distinct parts.

The three column products were combined in the product evaporator and treated with two, instead of the usual one, fuming HNO_3 separations. Following the transfer to the shipping cone, a rinse of the product evaporator revealed that about 7,500 curies were left in the evaporator tank, undoubtedly in the sulfate form. This material was again treated with fuming HNO_3 and all but 2,200 curies were recovered.

The equipment operated relatively well during this run. The only difficulties experienced which caused minor delays were the burning out of the clam shell heaters used to dry the product, the poor operation of the product evaporator tank sampler which made sampling almost impossible, and the plugging of the process filter during the last transfer of product from the extractor to the resin column cubicle.

The analytical summary of the run, corrected to last separation time 1804 on July 22, follows:

Slugs loaded	71 - 8" Hanford slugs
Slugs dissolved	69.7 - 8" Hanford slugs by analysis

[redacted]

[redacted]

This document contains restricted data as defined i[redacted]

	Curies	Percent
Product		
Product shipped (based on Los Alamos measurements)	22,800	61.59
Total losses	10,813	29.21
Material balance		90.80

E. J. Witkowski

E. J. Witkowski

EJW:hg

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X-426 (Revised 1-52)

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CENTRAL FILES NUMBER
55-4-10

DATE: April 4, 1955

SUBJECT: Report on RaLa Run No. 59

TO: A. F. Rupp

FROM: E. J. Witkowski

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TO A. F. Rupp
LOCATION

DATE April 4, 1955

ANSWERING LETTER DATE

ATTENTION
COPY TO M. E. Ramsey
F. L. Culler

SUBJECT Report on Rala Run No. 59

The run was started on March 1, completed on March 7 and shipped on March 8. The shipment contained approximately 20,000 curies according to our own measurements. Los Alamos however, reported only 11,000 curies. The discrepancy in measurements cannot be reconciled by waste analyses and a thorough check of the equipment even though losses of this magnitude are normally obvious or easily found.

In spite of the difference in product measurements, the run was considered successful because it satisfied the needs at Los Alamos until the latter part of April. The next run is tentatively scheduled to start on April 14.

No serious hazards were encountered during this run although there were two instances of minor air contamination during the dissolving operation.

The most serious equipment failure was the breakdown of the product cone manipulator. Fortunately, it occurred before the product was transferred to the cone so that the repairs could be made without excessive exposure of personnel. If the breakdown had occurred after the product transfer, it would have been impossible to perform repairs without discarding the entire product. The time lost for repairs was 8 hours.

Other equipment which was not operating during this run (because repairs were considered impractical) was the product tank sampler and the extractor agitator. Repair or replacement of the sampling valves in the resin column cubicle is an extremely expensive job and experience has shown that after these valves are repaired there is no assurance that they will operate for more than one run. The repair of the extractor agitator would require several months of shutdown which could not be justified, since an air sparger was found to be a satisfactory substitute for mechanical agitation.

The highest process loss (16.4%) was again experienced in the metatheses steps. The product was lost as a precipitate which appeared to be so fine that it passed through the sintered stainless steel process filters.

[redacted] [redacted]

[redacted]

The extraction loss (5.2%) was higher than normal as a result of an accidental transfer of 15 gallons of slurry to the wastetank during the fourth extraction. The material was transferred back to the extractor for recovery but the waste tank could not be thoroughly rinsed because of the limited capacity of the extraction tank. How and when the accidental transfer occurred could not be determined.

Other than the inoperability of the product sampler, which prevented an analytical check on the quantity of product, the resin column cubicle equipment performed relatively well.

Procedure changes incorporated in this run included: (1) return to using Dowex 50 resin after it was shown in the last run that Dowex 50 W resin did not eliminate discoloration of the product; (2) reduction in volume of the first metathesis by 10 gallons to prevent product loss by tank overflow which may have occurred in the last run; (3) the addition of inactive barium to the extraction to compensate for a deficiency resulting from a recent reduction in time exposure of uranium in the Hanford reactors.

The analytical summary of the run, corrected to L.S.T. 2330 on March 6, follows:

Slugs loaded	89 - 8"
Slugs dissolved	86.6 - 8" (by analysis)

	Curies	Percent
Product in UNH	34,697	100.00
Cell A losses		
Extraction wastes	1,816	5.23
First metathesis waste	4,331	12.48
Second metathesis waste	1,359	3.92
Extraction tank and process filter rinse	188	.54
Total	7,694	22.17
Resin Cubicle Feed		
Acetate feed	20,202	58.22
Versene feed	3,707	10.68
Total	23,909	68.90

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[redacted]

	Curies	Percent
Resin Cubicle Losses		
Versenate and NaOH wastes	92	0.27
HCl elution	1	0.00
$NaNO_3$ elution	10	0.03
Fuming HNO_3 precipitation	2,125	6.12
Feed tank rinse	43	0.12
Product evaporator rinse	325	0.94
Total	2,596	7.48
Product		
Based on L.A. measurements	11,100	31.99
Based on ORNL measurements	20,000	57.64
Total loss (by analyses)	10,290	29.65
Material balance (based on L.A. measurements)		61.65
Material balance (based on ORNL measurements)		87.29

E. J. Witkowski

E. J. Witkowski

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SUBJECT: Report on Rala Run No. 60

TO: A. F. Rupp

FROM: E. J. Witkowski

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H. R. C...
Chief, Declassification Branch

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David L Hamrin 2/3/95
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TO A. F. Rupp
LOCATION

DATE May 10, 1955

ANSWERING LETTER DATE

ATTENTION
COPY TO M. E. Ramsey
F. L. Culler

SUBJECT REPORT ON RALA RUN NO. 60

The run was started on April 14 and shipped on April 21. It contained 11,300 curies of product based on measurements at Los Alamos. Our own measurements, because of a recent change in instrumentation, were questionable and indicated either 12,300 or 20,000 curies. Therefore, it is presumed that Los Alamos measurements were essentially correct.

The quantity of product produced was disappointing and about 20,000 curies less than was reasonable to expect with losses incurred up to the product drying step in the shipping cone. During this operation the material escaped into the off-gas line either by overfilling the cone with rinses from the product evaporator tank or by sputtering during the drying operation. It was noted that the amount of precipitate in the cone was larger than usual and the sputtering action was great enough to throw out particles onto the viewing mirror when the product was viewed through the periscope. Overfilling of the cone, the other possible explanation of the loss, could have been caused by the erratic operation of the product evaporator tank liquid level instrument. Since this condition was not new in spite of routine checking of this instrument prior to every run, Los Alamos has agreed to try to mark and calibrate the cone in such a way as to permit reading of volumes directly inside of the cone between the transfers of rinses.

Even though the shipment was smaller than expected, the quantity satisfied the needs at Los Alamos for at least three months. A shipment of 500 curies (smallest in many years) is tentatively scheduled for the middle of July.

From the standpoint of safety and ease of operation, the run was relatively uneventful. The only difficulties encountered were slower than usual filtrations and one incident of above average plant wide air count that was not considered serious.

The analytical summary of the run, corrected to L.S.T. 1915 on April 20, follows:

Slugs loaded	84 - 8"
Slugs dissolved	84.4 - 8" (by analysis)

[redacted]

[redacted]

	Curies	Percent
Product in UNH	46,807	100.00%
Cell A Losses		
Extraction wastes	459	.98
First metathesis waste	2,495	5.33
Second metathesis and water wash	305	.65
Extraction tank and process filter rinse	485	1.04
Total	3,744	8.00
Resin Cubicle Feed		
Acetate Feed	39,328	84.02
Versene Feed	1,263	2.70
Total	40,591	86.72
Resin Cubicle Losses		
Versenate and NaOH waste	1,052	2.25
HCl elution waste	47	.10
Sodium Nitrate elution waste	140	.30
Fuming nitric precipitation	1,721	3.68
Feed tank rinse	19	.04
Product evaporator rinse	301	.64
Resin cubicle condensate	154	.33
Total	3,434	7.34
Product		
Product shipped (based on L.A. measurements)	11,300	24.14
Material balance		39.48

E J Witkowski

E. J. Witkowski

EJW:hg

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ORNL
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55-8-7

DATE: August 2, 1955

SUBJECT: Report on Rala Run No. 61

TO: A. F. Rupp

FROM: E. J. Witkowski

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TO A. F. Rupp
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August 2, 1955

ANSWERING LETTER DATE

ATTENTION
COPY TO M. E. Ramsey
F. L. Culler

SUBJECT Report on Rala Run No. 61

Approximately 700 curies of product was shipped on July 22. Radiation measurements made at ORNL were confirmed by Los Alamos. The quantity produced was 200 curies in excess of Los Alamos' request.

Only four 8 inch Hanford slugs were used. Since the dissolver capacity was too large (135 gal) for processing such a small amount of uranium, 50 X-slugs were also dissolved to obtain a proper operating volume. The X-slugs were old ones discharged into the canal after slug ruptures.

Thirty eight percent (882 curies) of the product contained in the slugs was lost in the dissolver heels. Being a one-batch run, the dissolving operation was very slow and was discontinued after 12 hours. The product lost would have had no value to the customer anyway since he could not possibly use any excess over 500 curies. Even the 200 curie excess he actually received was permitted to decay at Los Alamos before the product was used.

Another high loss (10%) experienced was in the sulfate extraction waste. The reason for the loss is not known; the metathesis filtration that follows the extraction filtration through the same filters gave a low loss (4%). Usually the metathesis loss is greater than the extraction loss.

The high loss encountered in the transfer of product to the shipping cone in the last several runs was eliminated in this run by minor changes in operating techniques.

The product drying operation required a much longer than normal time (27 hours) because of the very low product content. Most of the heat in a normal run is supplied by the product itself.

The analytical summary of the run follows:

Slugs loaded 4 - 8 inch Hanford
50 - X

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	Curies	Percent
Product in Dissolver	2,302	100.0
Cell A Losses*		
Sulfate extraction waste	223	9.7
First metathesis waste	48	2.1
Second metathesis and water wash	41	1.8
Dissolver heels	882	38.3
Total	1,194	51.9
Resin Cubicle Product		
Acetate feed	822	
Versene feed	37	
Total	859	37.3
Resin Cubicle Losses		
Feed effluent, Versenate, and NaOH waste	3	0.1
HCl elution	0	0.0
Sodium nitrate elution	3	0.1
Fuming nitric precipitation	33	1.4
Feed tank rinse	0	0.0
Product evaporator rinse	62	2.7
Total	101	4.3
Product		
Product shipped	700	30.4
Total losses*	1,295	56.3
Material balance		86.7

*Loss of product encountered in transfer out of extraction tank is not included in summary; because of operating difficulties, the extraction tank was not rinsed.

E J Witkowski

E. J. Witkowski

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55-10-12

DATE: October 6, 1955

SUBJECT: Report on Rala Run No. 62

TO: A. F. Rupp

FROM: E. J. Witkowski

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A. F. Rupp

October 6, 1955

M. E. Ramsey
F. L. Culler

Report on Rala Run No. 62

Between 18,000 and 20,000 curies were shipped to Los Alamos on September 3. The quantity was roughly twice the minimum amount required by the customer. The quality, as reported by Los Alamos, was very good.

Only one minor difficulty was experienced during this run. That was a back-up of radioactive solution into a steam jet line. The condition was corrected without overexposure of operating personnel.

The analytical results for the run follow with all values corrected to last separation time of 0900 on September 1, 1955.

Slugs loaded - 71 - 8" Hanford
Slugs dissolved - 67.6 - 8" Hanford

	Curies	Percent
Product in Dissolver	36,036	100.0
Cell A Losses		
Sulfate extraction waste	473	1.3
First metathesis waste	2,280	6.3
Second metathesis & water wash	753	2.1
Extraction tank & process filter rinse	197	.6
Dissolver heels	213	.6
Total	3,916	10.9
Resin Cubicle Feed		
Acetate feed	28,611	79.4
Versene feed	948	2.6
Total	29,559	82.0

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A. F. Rupp October 6, 1955

	Curies	Percent
Resin Cubicle Losses		
Feed effluent, versenate & NaOH waste	84	0.1
HCl elution	3	0.0
$NaNO_3$ elution	55	0.2
Fuming nitric precipitation No. 1	624	1.7
" " " " 2	286	0.8
Feed tank rinse	12	0.0
Product evaporator rinse	2,440	6.8
Total	3,444	9.6
Product		
Product shipped (based on L.A. measurements)	19,000	52.7
Total losses	7,360	20.4
Material balance		73.1

E. J. Witkowski

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SUBJECT: RaLa PRODUCTION - CALENDAR YEAR 1955

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FROM: A. F. Rupp and E. J. Witkowski

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RUNS 59-63

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RaLa PRODUCTION - CALENDAR YEAR 1955

A. F. Rupp and E. J. Witkowski

PRODUCTION

A total of 60,000 curies (Los Alamos measurements) was produced in four regular runs. A small run, yielding 700 curies, was also made for special testing purposes at Los Alamos. The quantity of product contained in each shipment was satisfactory to the customer. There were no failures this year. All batches were shipped on schedule.

As in previous years, the process yield was poor (see Table 1); the product shipped was only 37% of the amount (corrected for decay) contained in the slugs charged into the dissolver. The largest loss of product was incurred in the final product evaporation in the shipping cone after all the chemical processing was completed. Although it was not possible to analyze for this loss, it is estimated that it was approximately 30%.

A great number of "dummy" evaporation runs were made to improve the drying techniques in order to reduce this loss. Although these tests appeared successful, the performance could not be duplicated with the large quantity of activity contained in a normal run. The only possible solution to the problem appears to involve extensive redesign and rebuilding of the equipment. This move could not be justified in view of the present AEC plans to turn over the work to the Phillips Petroleum Company, Idaho Chemical Processing Plant, in 1956. It is more practical now to continue using more starting material to make up for high loss until the Laboratory is relieved of its responsibility for RaLa production.

A high over-all loss of product (12%) was again experienced in the carbonate metathesis cake filtrations by the fine precipitate going through the filter. This loss is not considered as serious as the loss in product evaporation, not only because it was lower but because it is more predictable and occurs at a stage in the process where it could be recovered when necessary.

The RaLa operation in 1955 was the most successful ever experienced from the standpoint of safety and ease of operation; unlike the previous years, no unusual difficulties were experienced.

EQUIPMENT

A great amount of maintenance work would be necessary if the equipment were scheduled for long-term operation. Since only two more runs are definitely scheduled, an effort is being made to avoid major repair work in the cells that would involve great expense and excessive personnel

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exposure. At the present time, the equipment that does not operate includes all thermocouples in the dissolver, the mechanical agitator in the extractor, and the sampling equipment on one product evaporator. The equipment that operates erratically (part time) includes the samplers on the other product evaporator and a resin cubicle feed tank. In addition to the equipment listed, there are several known leaks in the shielded piping that cannot be repaired without complete decontamination of the cells.

FUTURE PRODUCTION PLANS

The Laboratory is committed to produce only two more batches before the operation is discontinued; one run is scheduled in January, and the other in April. The AEC has requested that the ORNL equipment be kept in standby for several months after that, and possibly until January 1, 1957, during the start-up of full-scale runs at the Idaho plant. Construction of that plant was completed in November 1955, and the equipment was being tested at the end of the year.

MANPOWER REQUIREMENTS

By taking over new jobs during the last several years without an addition of manpower, the Chemical Separations Department has steadily decreased the amount of manpower normally devoted to the RaLa program. Because of the intermittent nature of the operation, it was more economical to do a large portion of operation on overtime than to maintain a full crew in standby between runs. A total of less than eight man-years was charged to the RaLa operation during 1955.

After the RaLa operation is discontinued, it is planned to use the present RaLa operating personnel for the operation of the Fission Product Plant. The Fission Product Plant operations are scheduled to start only a short time after the RaLa operation is discontinued. The schedule will allow for training of the people in the new operation and will eliminate the need for a lay-off of people experienced in that type of work.

TABLE 1

ANALYTICAL SUMMARY

	Amount of Activity (Curies)					Total Activity	
	Run 59	Run 60	Run 61	Run 62	Run 63	Curies	(%)
Dissolver starting material	34,697	46,807	2,302	36,036	45,200	165,042	100.0
Cell A losses							
Dissolver heels	0	0	882	213	2,878	3,973	2.4
Sulfate extraction filtrations	1,816	459	223	473	327	3,298	2.0
Metatheses filtrations	5,690	2,800	89	3,033	8,046	19,658	11.9
Extractor and filter rinse	188	485	(no rinse)	197	184	1,054	0.6
Total Cell A loss	7,694	3,744	1,194	3,916	11,435	27,983	16.9
Resin cubicle losses							
Fuming nitric filtration	2,125	1,721	33	910	4,318	9,107	5.5
Product evaporator rinse	325	301	62	2,440	277	3,405	2.1
All other	146	1,412	6	94	125	1,783	1.1
Total Cubicle loss	2,596	3,434	101	3,444	4,720	14,295	8.7
Total of all known losses	10,290	7,178	1,295	7,360	16,155	42,278	25.6
Product shipped (Los Alamos measurement)	11,100	11,300	700	19,000	19,000	61,100	37.0
Material balance (Percentage is given)	61.6	39.5	86.7	73.1	77.8	62.6	62.6
Estimated loss during product evaporation (not included in material balance)	10,000	25,000	50	7,000	7,500	49,550	30.0

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DATE: February 6, 1956

SUBJECT: Report on Rala Run No. 64

TO: A. F. Rupp

FROM: E. J. Witkowski

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OAK RIDGE NATIONAL LABORATORY

February 6, 1956

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To: A. F. Rupp

Subject: Report on Rala Run No. 64

A batch containing 16,300 curies (Los Alamos measurement) was shipped on January 20. The quantity of product was adequate to satisfy the customer's requirements until April 1956. The April run will be the last one scheduled at ORNL before production of Rala is turned over to the Phillips Petroleum Company's Idaho Chemical Processing Plant.

From the standpoint of ease of operation and safety, the run was as successful as any experienced to date. The only difficulty, also experienced in other runs and over which there was no control, was the high loss of product (approximately 12,000 curies) in the product drying step in the shipping cone.

The analytical results, with all values corrected to last separation time 0620 on January 19, follow:

Slugs loaded - 100 - 8" Hanford
Slugs dissolved - 93. (by analysis)

	Curies	Percent
Product in Dissolver	49,124	100.0
Cell A Losses		
Sulfate extraction waste	2,612	5.3
First metathesis waste	5,245	10.7
Second metathesis and water wash	1,465	3.0
Extraction tank and process filter rinse	89	0.2
Dissolver heels	7,794	15.9
Total	17,205	35.1
Resin Cubicle Feed		
Acetate feed	29,874	60.8
Versene feed	938	1.9
Total	30,812	62.7

	Curies	Percent
Resin Cubicle Losses		
Feed effluent, versenate and NaOH Losses	284	0.6
HCl elution	5	0.0
$NaNO_3$ elution	61	0.1
Fuming nitric precipitation	1,936	3.9
Feed tank rinse	31	0.1
Product evaporator rinse	223	0.5
Total	2,540	5.2
Product		
Product shipped	16,300	33.2
Total losses	19,745	40.2
Material balance*		73.4

*Does not include the estimated loss of approximately 12,000 incurred in the product drying step. With this loss included, the material balance would be about 98%.

E. J. Witkowski

E. J. Witkowski

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cc: MERamsey
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ORNL
CENTRAL FILES NUMBER
56-4-63

This document consists of 2 pages.
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DATE: April 12, 1956

SUBJECT: Report on Rala Run No. 65

TO: A. F. Rupp

FROM: E. J. Witkowski

FILE Rala

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X-847

INTRA-LABORATORY CORRESPONDENCE

OAK RIDGE NATIONAL LABORATORY

April 12, 1956

Classification Cancelled
~~Or Changed To~~
By Authority Of DOC
By EEB Date SEP 7 1971

To: A. F. Rupp

Subject: Report on Rala Run No. 65

Approximately 17,500 curies of product was shipped to Los Alamos on March 28. No operating difficulties were encountered although between 7,000 and 10,000 curies of product was again lost into the off-gas line during the drying operation in the shipping cone. A change in the drying procedure, which allowed the product to dry by its own heat, was not effective in reducing the high loss regularly encountered in this operation.

The analytical summary, with all values corrected to Last Separation Time 0620 on March 26, follows:

Slugs loaded - 71 - 8" (Hanford)
Slugs dissolved - 71

	Curies	Percent
Product in Dissolver	54,662	100.0%
Cell A Losses		
Sulfate extraction waste	497	0.9
First metathesis waste	4,580	8.4
Second metathesis waste	662	1.2
Extraction tank and process filter rinse	678	1.2
Dissolver heels (not used)	12,032	22.0
Total	18,449	33.7
Resin Cubicle Feed		
Acetate feed	34,800	63.7
Versene feed	700	1.3
Total	35,500	65.0
Resin Cubicle Losses		
Feed effluent, versenate & NaOH losses	94	0.2
HCl elution	2	0.0
$NaNO_3$ elution	136	0.2
Fuming nitric precipitation	4,130	7.6
Feed tank rinse	952	1.7
Product evaporator rinse	1,070	2.0
HNO_3 column rinse	388	0.7
Total	6,772	12.4

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[redacted]

A. F. Rupp

April 12, 1956

	Curies	Percent
Product		
Product shipped (based on L.A. measurement)	17,423	31.9
Total of all losses shown by analysis	25,221	46.1
Material balance*		78.0

*Does not include the 7,000 - 10,000 curie loss incurred in the product drying step.

The next run is tentatively scheduled for the early part of May. The May run will be the third one in calendar year 1956 and the sixth one in the present fiscal year.

EJWitkowski

E. J. Witkowski

EJW:hg
cc: MERamsey
FLCuller

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ORNL
CENTRAL FILES NUMBER
56-6-49

DATE: June 11, 1956

SUBJECT: Report on Kala Run No. 66

TO: A. F. Rupp

FROM: E. J. Witkowski

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OAK RIDGE NATIONAL LABORATORY

June 11, 1956

To: A. F. Rupp

Subject: Report on Rala Run No. 66

The run was started on May 7 and completed on May 12. The shipment contained 18,000 curies of product as measured at ORNL; Los Alamos measurements indicated 15,600 curies.

The operations were performed with only minor difficulties. The extraction filtration losses were higher than normal and it was necessary to settle and decant the second extraction waste to recover 3,000 curies of a 3,500 curie loss. Approximately 8.4% of the product could not be accounted for analytically after transfer from cell A to the resin cubicle.

A change in the product drying procedure, using a lower temperature and trying to maintain a constant vacuum, did not reduce the usual high loss encountered in this operation; 8,000 curies were lost.

The analytical summary, with all values corrected to last separation time 0300 on May 12, follows:

Slugs loaded - 87 - 8" (Hanford)
Slugs dissolved - 85.3 - 8"

	Curies	Percent
Product in Dissolver	50,900	100.0%
Cell A Losses		
Sulfate extraction waste	1,954	3.8
First metathesis waste	6,033	11.9
Second metathesis waste	1,727	3.4
Extraction tank and process filter rinse	892	1.8
Dissolver heels (not used)	6,727	13.2
Total	17,333	34.1
Resin Cubicle Feed		
Acetate feed	27,429	53.9
Versene feed	1,852	3.6
Total	29,281	57.5

[redacted]

	Curies	Percent
Resin Cubicle Losses		
Feed effluent, versenate & NaOH Losses	440	0.9
HCl elution	7	0.0
$NaNO_3$ elution	131	0.3
Fuming nitric precipitation	4,104	8.1
Feed tank rinse	64	0.1
Product evaporator rinse	315	0.6
	5,061	10.0
Product		
Product shipped (based on L.A. measurements)	15,600	30.6
Total of all losses shown by analyses	22,394	44.0
Material balance*		74.6

*Does not include estimated loss of 8,000 curies incurred in product drying step.

The next run is tentatively scheduled for September. It will be the fourth in calendar year 1956.

E J Witkowski

E. J. Witkowski

EJW:hg
cc: MERamsey
FLCuller

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ORNL
CENTRAL FILES NUMBER
56-9-104

DATE: September 27, 1956

SUBJECT: Report on Rala Run No. 67

TO: H. E. Seagren

FROM: E. J. Witkowski

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LABORATORY CORRESPONDENCE

OAK RIDGE NATIONAL LABORATORY

September 27, 1956

Classification Cancelled
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By Authority Of DOC
By EEB Date SEP 7 1971

To: H. E. Seagren

Subject: Report on Rala Run No. 67

On September 9, 1956, Rala Run No. 67 was started with the loading of 130 eight inch Hanford slugs. The run was completed on September 14, 1956 with a shipment of 13,750 curies of product as measured by radiation readings at ORNL. Measurements at Los Alamos indicated that the shipment contained 14,900 curies.

Los Alamos at first expressed satisfaction over the quantity of the product produced but ten days later said that a shortage was experienced because of their own scheduling difficulties.

The operations proceeded normally through the extraction steps with very low losses. During the large (100 gallon) metathesis step, approximately 20% of the metathesis volume was lost due to a syphoning action through the overflow line. The lost material was discarded to the tank farm before the volume discrepancy was noted. The run proceeded smoothly through the final steps without incident. The loss from the fuming nitric precipitation step was double the normal loss and an investigation is under way to determine if the product evaporator tank filter is at fault.

CLASSIFICATION CANCELLED
ADD signature: [signature] Date: 1/19/95
Single rereview of CCRP-declassified documents was authorized by DOE Office of Declassification memo of August 22, 1994

The analytical summary, corrected to an L.S.T. of 1100 on September 13, 1956 is as follows:

Slugs loaded - 130 - 8" Hanford
Slugs dissolved - 129.2

	Curies	Percent
Curies dissolved	48,050	100.0
Cell A Losses		
Sulfate extraction waste	535	1.1
Metathesis	7,124	14.8
Tank rinses	140	0.3
Heel dissolving	4,780	9.9
Total	12,579	26.1
Resin Cubicle Feed		
Acetate feed	27,800	57.9
Versene feed	1,070	2.2

[redacted]

September 27, 1956

	Curies	Percent
Resin Cubicle Losses		
Elution wastes	19	0.0
Tank rinses	847	1.8
Fuming nitric waste	8,950	18.6
Total	9,816	20.4
Product		
Product shipped (based on L.A. measurements)	14,900	31.0
Total of all losses shown by analyses	22,395	46.6
Material balance		77.6

Following the run shipment, all equipment was decontaminated and rinsed to place it in readiness for the next run which is tentatively scheduled to start on October 20. It is expected that this will be the last run scheduled for this year and possibly the final run before the operation is transferred to Idaho.

E. J. Witkowski

RWS:hg
cc: MERamsey
FRCuller

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11/14/56

ORNL
CENTRAL FILES NUMBER
56-11-28

DATE: November 9, 1956

SUBJECT: Report of Rala Run No. 68

TO: H. E. Seagren

FROM: E. J. Witkowski

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[redacted]

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D. McGhee
For: N. T. Bray, Supervisor
Laboratory Records Dept.
ORNL

This document has been approved for release to the public by:

David R Hamrin 3/1/95
Technical Information Officer
ORNL Site
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X-847

INTRA-LABORATORY CORRESPONDENCE

OAK RIDGE NATIONAL LABORATORY

November 9, 1956

To: H. E. Seagren

Subject: Report on Rala Run No. 68

A shipment of 15,000 curies was made to Los Alamos on October 26. The run was started on October 21 and completed on October 25 with only few operating difficulties.

The main trouble occurred in product measurement, after all processing was completed. The measuring instruments failed and all efforts to repair them, before it was necessary to remove the product carrier from the loading cubicle, were unsuccessful. At the time of shipment, it was estimated that the product contained between 10,000 and 17,000 curies. Los Alamos later reported that the shipment contained 15,000 curies.

Approximately 45% of the product (excluding decay) could not be accounted for by chemical analysis. One half of this was lost in Cell A, during the extraction and metathesis operation, the balance, as usual, during the product evaporation step. An analytical summary of the run, corrected to last separation time 0010 on October 25 follows:

Slugs loaded - 70 - 8" Hanford
Slugs dissolved - 71.8

	Curies	Percent
Curies dissolved	47,869	100.0
Cell A Losses		
Sulfate extraction waste	725	1.5
Metathesis waste	3,472	7.3
Tank rinses	969	2.0
Unused dissolver heels	4,170	8.7
Total	9,336	19.5
Resin Cubicle Feeds		
Acetate feed	22,308	46.6
Versene feed	5,542	11.6
Total	27,850	58.2

[redacted]

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[redacted]

	Curies	Percent
Resin Cubicle Losses		
Elution wastes	270	0.6
Tank rinses	893	1.8
Fuming nitric waste	1,010	2.1
Total	2,173	4.5
Product		
Product shipped	15,000	31.4
Total of all losses shown by analyses	11,509	24.0
Material balance		55.4

E J Witkowski

E. J. Witkowski

EJW:hg
cc: AFRupp
FLCuller

[redacted]

[redacted]

	Curies	Percent
Resin Cubicle Losses		
Elution wastes	270	0.6
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Fuming nitric waste	1,010	2.1
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E J Witkowski

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56-11-28

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DATE: November 9, 1956

SUBJECT: Report of Rala Run No. 68

TO: H. E. Seagren

FROM: E. J. Witkowski

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For: N. T. Bray, Supervisor
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INTRA-LABORATORY CORRESPONDENCE

OAK RIDGE NATIONAL LABORATORY

November 9, 1956

To: H. E. Seagren

Subject: Report on Rala Run No. 68

A shipment of 15,000 curies was made to Los Alamos on October 26. The run was started on October 21 and completed on October 25 with only few operating difficulties.

The main trouble occurred in product measurement, after all processing was completed. The measuring instruments failed and all efforts to repair them, before it was necessary to remove the product carrier from the loading cubicle, were unsuccessful. At the time of shipment, it was estimated that the product contained between 10,000 and 17,000 curies. Los Alamos later reported that the shipment contained 15,000 curies.

Approximately 45% of the product (excluding decay) could not be accounted for by chemical analysis. One half of this was lost in Cell A, during the extraction and metathesis operation, the balance, as usual, during the product evaporation step. An analytical summary of the run, corrected to last separation time 0010 on October 25 follows:

Slugs loaded - 70 - 8" Hanford
Slugs dissolved - 71.8

	Curies	Percent
Curies dissolved	47,869	100.0
Cell A Losses		
Sulfate extraction waste	725	1.5
Metathesis waste	3,472	7.3
Tank rinses	969	2.0
Unused dissolver heels	4,170	8.7
Total	9,336	19.5
Resin Cubicle Feeds		
Acetate feed	22,308	46.6
Versene feed	5,542	11.6
Total	27,850	58.2

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	Curies	Percent
Resin Cubicle Losses		
Elution wastes	270	0.6
Tank rinses	893	1.8
Fuming nitric waste	1,010	2.1
Total	2,173	4.5

	Curies	Percent
Product		
Product shipped	15,000	31.4
Total of all losses shown by analyses	11,509	24.0
Material balance		55.4

E. J. Witkowski

E. J. Witkowski

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ORNL
CENTRAL FILES NUMBER
57-4-16

RUNS
re: 64-68

DATE: April 8, 1957

SUBJECT: RaLa PRODUCTION - CALENDAR YEAR 1956

TO: Listed Distribution

FROM: E. E. Seagren and E. J. Witkowski

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ORNL RaLa PRODUCTION - CALENDAR YEAR 1956

H. E. Seagren and E. J. Witkowski

RaLa production at ORNL was concluded at the end of calendar year 1956. This report summarizes the production experience for this year.

PRODUCTION

Six batches of RaLa product containing a total of 79,000 curies were shipped to Los Alamos during CY-1956. The quantity produced in each run was satisfactory to the customer although, as in previous years, additional purification was necessary at Los Alamos to remove impurities from the product. All runs were shipped on schedule; there were no failures.

The average shipment in CY-1956 contained 15,840 curies as compared to 15,100 curies (excluding one special 700-curie run) in CY-1955. Total processing losses increased from 63.0% in CY-1955 to 68.4% in CY-1956 because of extra uranium loaded into the dissolver for standby in case of an abnormal product loss due to equipment failure. The need for the additional starting material, however, never materialized. Consistent with past experience, the largest single process loss occurred in the final product evaporation step although the estimated amount was decreased this year from 30% to 17%. The decrease is attributed to better operating techniques and modifications in the drying procedure, developed after extensive testing with nonradioactive solutions. An analytical summary for the runs made this year is shown in the attached table.

Contrary to all expectations, the extremely poor condition of the equipment created no operating difficulties; in fact, operations in CY-1956 as well as in CY-1955, were almost completely trouble-free. Radiation exposure of operating personnel was also at the lowest level in the ORNL RaLa production experience.

EQUIPMENT

As in CY-1955, extensive maintenance that would be required for long term operations was not attempted in order to avoid exposure of operating and maintenance forces and to reduce expenditures. At the beginning of CY-1956, the Oak Ridge National Laboratory was scheduled definitely for only two more runs before going out of the RaLa production although, later, three additional runs were requested.

The equipment inoperable at the end of CY-1956 includes all thermocouples in the dissolver, the mechanical agitator in the extractor and samplers on both product evaporators. The other sampling equipment works only part of the time. No samples for analysis of the product could be taken in any run prior to shipment. The radiation product measuring instrument which usually gives a fair indication of the product quantity also failed completely during the last run just prior to the removal of the product in the shipping container. To avoid spreading contamination while repairing the instrument, the shipment was made with only a rough estimate of quantity; Los Alamos later reported receiving 15,000 curies.

CONCLUSION OF RaLa PRODUCTION

The Laboratory was officially relieved of its RaLa production responsibilities at the end of CY-1956. The AEC requested that the Laboratory maintain the present equipment in standby until March 31, 1957, during the initial runs in new facilities in Idaho.

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ANALYTICAL SUMMARY

	Amount of Activity (Curies)					Total Activity	
	Run 64	Run 65	Run 66	Run 67	Run 68	Curies	(%)
Dissolver starting material	49,124	54,662	50,900	48,050	47,869	250,605	100.0
Cell A losses							
Unused dissolver heels	7,794	12,032	6.727	4,780	4,170	35,503	14.2
Sulfate extraction filtrations	2,612	497	1,954	535	725	6,323	2.5
Metatheses filtrations	6,710	5,242	7,760	7,124	3,472	30,308	12.1
Extractor and filter rinses	89	678	892	140	969	2,768	1.1
Total Cell A loss	17,205	18,449	17,333	12,579	9,336	74,902	29.9
Resin cubicle losses							
Fuming nitric filtrations	1,936	4,130	4,104	8,950	1,010	20,130	8.0
Product evaporator and feed tank rinse	254	2,022	379	847	893	4,395	1.8
All other	350	620	578	19	270	1,837	0.7
Total Cubicle loss	2,540	6,772	5,061	9,816	2,173	26,362	10.5
Total of all losses shown by analyses	19,745	25,221	22,394	22,395	11,509	101,264	40.4
Product shipped (measured by Los Alamos)	16,300	17,400	15,600	14,900	15,000	79,200	31.6
Material balance (percentage is given)	73.4	78.0	74.6	77.6	55.4		72.0
Estimated product drying loss (not included in material balance)	12,000	9,000	8,000	4,000	10,000	43,000	17.2

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